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FOREWORD

The *Proceedings* of the American Society for Testing and Materials are published annually and include all reports and papers offered to the Society during the current year and accepted by the Administrative Committee on Papers and Publications for the *Proceedings,* together with discussion.

The table of contents and subject and author indexes cover all papers and reports published by the Society during the current year, which in addition to those appearing in the *Proceedings* include those accepted for publication in *Materials Research & Standards* or in Special Technical Publications.

A list of the Special Technical Publications published by the Society in 1961 is given on page 1469 of this volume. This supplements the lists appearing in the *Proceedings* from 1948 to 1960 covering all special publications published by the Society up to and including 1960.

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SUMMARY OF PROCEEDINGS

OF THE

SIXTY-FOURTH ANNUAL MEETING

ATLANTIC CITY, N. J., JUNE 25–30, 1961

This summary of the Sixty-fourth Annual Meeting of the American Society for Testing Materials, held at the Chalfonte-Haddon Hall Hotel, Atlantic City, N. J., June 25–30, 1961, is a record of the transactions of the meeting, including the actions taken on the various recommendations submitted by the technical committees. In all, 41 technical sessions were held.

The registered attendance of the meeting is as follows: Members present or represented, 1865; committee members, 1205; guests, 392; total, 3462; ladies, 430.

The Proceedings are set forth session by session. There were 81 reports and 118 formal papers presented. The record with respect to each has been briefed; the recommendations in the reports have been grouped so as to cover the acceptance of material for publication as tentative, such as new specifications, methods of test, revisions of tentatives, and proposed revisions of existing standards, and, as a separate group, the approval of matters that were referred to letter ballot of the Society, comprising the adoption of tentatives as standard, the adoption of revisions of standards, and the immediate adoption as standard without prior publication as standard. Accordingly, wherever the action is reported "adopted as standard" or "adopted as standard, revisions in," it is understood that this indicates approval of the Annual Meeting for reference to letter ballot of the Society.[1] The various recommendations so recorded are included in the Society letter ballot. The actions designated as "accepted as tentative" or "accepted as tentative, revisions in" are self-evident as indicating acceptance by the Society at the Annual Meeting for publication as tentative. Designations that have since been assigned to new tentatives are included as information.

All papers, unless otherwise noted, are published in the *Proceedings*.

While all the items on the program are recorded under the particular session in which they are presented, for convenience in locating actions with respect to any particular report, the accompanying list is presented of all reports together with the page reference where the actions thereon are recorded.

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[1] The letter ballot on recommendations affecting standards, distributed to the Society membership, will be canvassed on September 18, 1961. See Editor's Note, p. 44.

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FIRST SESSION—OPENING SESSION

MONDAY, JUNE 26, 9:15 A.M.

FORMAL OPENING OF THE SIXTY-FOURTH ANNUAL MEETING, PRESIDENT A. ALLAN BATES

The session was formally opened by President A. Allan Bates, who called attention to the very complete program of events that were to follow during the ensuing week. The meeting was then turned over to the session chairman, H. J. Grover, Battelle Memorial Inst.

SESSION ON FATIGUE

SESSION CHAIRMAN: H. J. GROVER

A Proposed New Relation for Cumulative Fatigue Damage in Bending—S. S. Manson, A. J. Nachtigall, and J. C. Freche, presented by Mr. Manson.

A Study of the Accumulation of Fatigue Damage in Steel—W. H. Erickson and C. E. Work, presented by Mr. Erickson.

Effect of Loading Sequence on Cumulative Fatigue Damage of 7075-T6 Aluminum Alloy—Robert Spitzer and H. T. Corten, presented by Mr. Corten.

A Relation Between Theoretical Stress Concentration Factor and Fatigue Notch Factor Deduced from the Concept of Highly Stressed Volume—Roberto Kuguel, presented by H. T. Corten.

(*Continued in Tenth Session*)

SECOND SESSION—SYMPOSIUM ON SOIL DYNAMICS[2]

MONDAY, JUNE 26, 9:30 A.M.

SESSION CHAIRMAN: ROCKWELL SMITH

Testing Procedures for Model Footings and Presentation of Tradex Data—J. A. Alai, presented by the author.

On Biaxial Stress Field in Non-Cohesive Soils Subjected to Vibratory Loads—R. K. Bernhard, presented by the author.

Stress-Deformation Relations for Soft Saturated Silt Under Low Frequency Oscillating Direct Shear Forces—F. J. Converse, presented by the author.

Dynamic Loading Machine and Results of Preliminary Small-Scale Footing Tests—R. W. Cunny and R. C. Sloan, presented by Mr. Sloan.

Performance of Embedded Pressure Gages Under Static and Dynamic Loadings—A. J. Durelli and W. F. Riley, presented by Mr. Riley.

Bearing Capacities of Dynamically Loaded Footings—S. Shenkman and K. E. McKee, presented by Mr. McKee.

Facilities for Dynamic Testing of Soils—G. K. Sinnamon and H. M. Newmark, presented by Mr. Sinnamon.

The Damping Capacity of Some Granular Soils—G. F. Weissmann and R. R. Hart, presented by Mr. Weissmann.

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[2] Issued as *ASTM STP No. 305.*

APPLIED STATISTICS IN ASTM WORK

MONDAY, JUNE 26, 9:30 A.M.

The following papers were presented at a session sponsored by Subcommittee I on ASTM Problems of Committee E 11 on Quality Control.

Introduction by C. A. Bicking.

Recommended Procedure for Conducting an Interlaboratory Study of a Test Method—Grant Wernimont.

Recommended Practice on Definitions of the Terms Precision and Accuracy as Applied to Measurement of a Property of a Material—R. B. Murphy. (Since accepted for publication as Recommended Practice E 177 - 61 T).

Question Period.

TECHNICAL SESSION ON ELECTRON METALLOGRAPHY

MONDAY, JUNE 26, 9:30 A.M.

The following papers were presented at an informal session sponsored by Subcommittee XI on Electron Metallography of Metals of Committee E-4 on Metallography.

Progress Report by Task Force D, Subcommittee XI, on Round Robin Testing of an Nb-Fe Diffusion Couple by Electron Probe Analysis Methods—A. W. Danko.

X-Ray Diffraction in the Electron Probe Microanalyzer—B. H. Heise.

A Study of the Microstructures of Precipitation Hardening Stainless Steel—J. C. Wilkins and R. E. Pence.

Nitride Morphology in 410 Stainless Steel—B. R. Banerjee, J. M. Capenos, and J. J. Hauser.

Film Entitled Motion of Dislocations in Aluminum Observed Directly Under the Electron Microscope—A. Berghezan, A. Fourdeux, and L. E. Pellier.

An Electron Microscopic Investigation of the Tensile Deformation of an Age Hardened Nickel-Base Alloy—G. I. Madden, W. C. Bigelow, and D. L. Sponseller.

Multiple Reflection Phenomena in Electron Diffraction—H. J. Beattie, Jr.

Direct Transmission Electron Microscopy of Zirconium—A. P. Young, C. W. Melton, and C. M. Schwartz.

Transmission Electron Metallography of the ASTM Eutectoid Steel—G. E. Pellissier.

THIRD SESSION—SYMPOSIUM ON MAJOR EFFECTS OF MINOR CONSTITUENTS ON THE PROPERTIES OF MATERIALS[3]

MONDAY, JUNE 26, 2:00 P.M.

SESSION CHAIRMAN: I. V. WILLIAMS

Impurity Effects in High-Purity Metals—G. A. Moore and L. L. Wyman, presented by Mr. Wyman.

The Effect of Impurities on the Properties of Ceramic Materials—I. B. Cutler, presented by the author.

Major Effects from Minor Constituents—Role in Organic Material Structures—J. F. Lontz, presented by the author.

(*Continued in Sixth Session*)

FOURTH SESSION—COMMITTEE REPORT SESSION

MONDAY, JUNE 26, 4:30 P.M.

SESSION CHAIRMAN: M. A. PINNEY

Committee A-2 on Wrought Iron:

Report presented in the absence of the chairman by O. M. Tishlarich, secretary, and the following action taken:

Adopted as Standard:

Spec. for Electric-Fusion (Arc)-Welded Wrought Iron Plate Pipe (A 419 – 57 T).

Committee A-9 on Ferro-Alloys:

Report presented by S. W. Poole, chairman, and the following actions taken:

Adopted as Standard:

Spec. for Ferromanganese (A 99 – 57 T)
Spec. for Ferromolybdenum (A 132 – 59 T)

Committee B-1 on Wires for Electrical Conductors:

Report presented in the absence of the chairman by C. E. Topping, vice-chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Test for Electrical Conductivity by Use of Eddy Currents (B 342 – 60 T)
Def. of Terms Relating to Uninsulated Metallic Electrical Conductors (B 354 – 60 T)

Adopted as Standard, Revisions in:

Test for Resistivity of Electrical Conductor Materials (B 193 – 60)

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[3] Issued as *ASTM STP No. 304*.

Spec. for Rolled Aluminum Rods (EC Grade) for Electrical Purposes (B 233 – 60)

Spec. for Standard Nominal Diameters and Cross-Sectional Areas of Awg Sizes of Solid Round Wires Used as Electrical Conductors (B 258 – 57)

Spec. for Three-Quarter Hard Aluminum Wire for Electrical Purposes (B 262 – 56)

Spec. for Half-Hard Aluminum Wire for Electrical Purposes (B 323 – 60)

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys:

Report presented in the absence of the chairman by W. H. Ailor, secretary, and the following actions taken:

Accepted as Tentative:

Method of Copper-Accelerated Acetic Acid - Salt Spray (Fog) Testing (Cass Test) (B 368 – 61 T)

Adopted as Standard:

Method of Salt Spray (Fog) Testing (B 117 – 57 T)

Method of Acetic Acid - Salt Spray (Fog) Testing (B 287 – 57 T)

Committee B-8 on Electrodeposited Metallic Coatings and Related Finishes:

Report presented by C. H. Sample, chairman, and the following actions taken:

Accepted as Tentative:

Specs. for Electrodeposited Coatings of Multi-Layer Nickel Plus Chromium on Steel (B 375 – 61 T)

Def. of Terms Relating to Electroplating (B 374 – 61 T)

Accepted as Tentative, Revisions in:

Spec. for Electrodeposited Coatings of Nickel and Chromium on Steel (A 166 – 58 T)

Adopted as Standard, Revisions in:

Specs. for Electrodeposited Coatings of Nickel and Chromium on Zinc and Zinc-Base Alloys (B 142 – 59)

Committee B-9 on Metal Powders and Metal Powder Products:

Report presented by J. L. Bonanno, chairman, and the following actions taken:

Accepted as Tentative:

Test for Density of Sintered Metal Friction Materials (B 376 – 61 T)

Test for Transverse Rupture Strength of Sintered Metal Friction Materials (B 378 – 61 T)

Specs. for Steel Backed Metal Powder Bearing and Bushing Alloys (B 377 – 61 T)

Accepted as Tentative, Revisions in:

Spec. for Copper-Infiltrated Iron Sintered Metal Powder Structural Parts (B 303 – 58 T)

Test for Compressibility of Metal Powders (B 331 – 58 T)

Adopted as Standard:

Test for Apparent Density of Refractory Metals and Compounds by the Scott Volumeter (B 329 – 58 T)

Adopted as Standard, Revisions in:

Specs. for Iron Copper Sintered Metal Powder Structural Parts (B 222 – 58)

Def. of Terms Used in Powder Metallurgy (B 243 – 58)

Spec. for Bronze Sintered Metal Powder Structural Parts (B 255 – 60)

Committee D-3 on Gaseous Fuels:

Report presented in the absence of the chairman by K. R. Knapp, secretary, and accepted as a report of progress.

Committee E-17 on Skid Resistance:

Report presented by E. A. Whitehurst, chairman, and accepted as a report of progress.

FIFTH SESSION—MARBURG LECTURE[4]

MONDAY, JUNE 26, 5:00 P.M.

SESSION CHAIRMAN: PRESIDENT A. ALLAN BATES

A brief statement of the purpose of the lecture was given by President Bates. Bruce Chalmers, Gordon McKay Professor of Metallurgy, Harvard University, was then introduced by President Bates as the thirty-fifth Edgar Marburg Lecturer. Speaking on the subject "The Nucleation and Growth of Ice Crystals," Mr. Chalmers discussed the theory of static nucleation. The process of crystal growth from the melt was examined in terms of the structure of the solid-liquid interface and thermal and chemical conditions. Some experiments on the effect of the temperature and the composition of aqueous solutions on the growth of ice crystals was described, and the differences between growth within a liquid and growth on the surface of a solid was accounted for. The subject of this lecture has direct implications on the freezing of water in soils, on the manufacture of semiconductors, on zone melting processes, and a host of other applications in the electronics industry and in metallurgy.

President Bates expressed appreciation to Mr. Chalmers for his excellent lecture and presented to him on behalf of the Society the Edgar Marburg Lecture Certificate.

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[4] Issued as a separate publication.

ROUND TABLE SESSION ON LOW-TEMPERATURE PROPERTIES OF NON-METALLIC MATERIALS

MONDAY, JUNE 26, 2:00 P.M.

This round table session was sponsored by the Panel on Structural Materials for Airframes and Missiles of the Joint Committee on Effect of Temperature on the Properties of Metals.

MODERATOR: J. WATSON, Convair Astronautics.

Speakers: J. Hertz, Convair Astronautics.
B. L. Taft, Pratt and Whitney.
R. L. Hauser, The Martin Co.
R. E. Mowers, Rocketdyne.

SIXTH SESSION—SYMPOSIUM ON MAJOR EFFECTS OF MINOR CONSTITUENTS ON THE PROPERTIES OF MATERIALS[3]

(Continued from the Third Session)

MONDAY, JUNE 26, 8:00 P.M.

SESSION CHAIRMAN: J. D. SULLIVAN

The Problem of Gas Purity in Plasma Research and Technology—S. J. Buchsbaum and G. A. Sawyer, presented by Mr. Buchsbaum.

Effects of Small Amounts of Extraneous Materials on the Properties of Petroleum, Petroleum Products, and Related Liquids—H. M. Smith, presented by the author.

SEVENTH SESSION—SESSION ON SOILS

MONDAY, JUNE 26, 8:00 P.M.

SESSION CHAIRMAN: C. R. FOSTER

C. A. Hogentogler Award:

The Sixth C. A. Hogentogler Award was made to C. B. Crawford, National Research Council of Canada, for his paper on "The Influence of Rate of Strain on Effective Stresses in Sensitive Clay," published in Papers on Soils, 1959 Meeting, *ASTM STP 254.*

Papers:

Some Observations on the Measurement of Sensitivity of Clays—W. J. Eden and J. K. Kubota, presented by R. F. Leggett.

The Influence of Strain on Shearing Resistance of Sensitive Clay—C. B. Crawford, presented by the author.

A Nomographic Procedure for the Detection of Suspect Unconfined Compressive Strength Values in a Series of Soil-Additive Strength Determinations—H. T. David, D. T. Davidson, and C. A. O'Flaherty, presented by Mr. O'Flaherty.[5]

Quantitative Determination of Soil Montmorillonite by X-ray Diffraction—G. R. Glenn and R. L. Handy, presented by Mr. Glenn.

A Study of Artificial Soils—E. T. Selig and R. D. Rowe, presented by Mr. Selig.

Standard Laboratory Control for Soil-Cement Mixtures—color film presented by Miles D. Catton.

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[5] Published in *Materials Research & Standards,* Vol. 1, No. 12, p. 947, Dec., 1961.

EIGHTH SESSION—SESSION ON CONCRETE

MONDAY, JUNE 26, 8:00 P.M.

SESSION CHAIRMAN: R. H. BRINK

The Indirect Tension Test for Concrete—N. B. Mitchell, Jr., presented by the author.[6]

Correlation of Flexural and Compressive Strengths of Concrete and Mortars—K. E. Palmer and I. L. Lynn, presented by Mr. Lynn.

Strain Distribution in Compressively Loaded Concrete Specimens—J. R. Keeton, presented by the author.

Methods for Determining Mechanical Resonance Frequencies and for Calculating Elastic Moduli from These Frequencies—S. Spinner and W. E. Tefft, presented by Mr. Tefft.

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[6] Published in *Materials Research & Standards,* Vol. 1, No. 10, p. 780, Oct., 1961.

(*Continued in Eighteenth Session*)

NINTH SESSION—SYMPOSIUM ON EVALUATION OF METALLIC MATERIALS IN DESIGN FOR LOW-TEMPERATURE SERVICE[7]

TUESDAY, JUNE 27, 9:00 A.M.

SESSION CHAIRMAN: F. B. FOLEY
CO-CHAIRMAN: G. R. IRWIN

Analysis of the Effects of Test Temperature on the Notch Strength of High-Strength Sheet Alloys—Volker Weiss and J. G. Sessler, presented by Mr. Weiss.

Welded Ferritic Steel Construction for Intermediate Low-Temperature Service: Considerations Arising from Tension Tests on Welded and Notched Wide Plates—A. A. Wells, presented by the author.

Effect of U-Notch Depth on Impact Resistance Under Simple Beam Loading—D. C. Reymond, presented by the author.

The Effect of Small Cracks on the Load-Carrying Ability of High-Strength Steel—G. K. Manning, presented by the author.

Fracture of High-Strength Sheet Steel Specimens Containing Small Cracks—J. E. Srawley and C. D. Beachem, presented by Mr. Srawley.

Tensile and Impact Properties of Cast Stainless Steels at Cryogenic Temperatures—E. R. Hall (presented by title only)

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[7] Issued as *ASTM STP No. 302.*

(*Continued in Thirteenth and Twenty-second Sessions*)

TENTH SESSION—SESSION ON FATIGUE

(Continued from the First Session)

TUESDAY, JUNE 27, 9:30 A.M.

SESSION CHAIRMAN: F. B. STULEN

Effect of Oleophobic Films on Fatigue Crack Propagation—W. L. Holshouser and H. P. Utech, presented by Mr. Holshouser.

A Fatigue Test for Printed Wiring Boards and Through Connections—G. R. Gohn and A. Fox, presented by Mr. Fox.[8]

Fatigue Properties of Uncoated and Coated Unalloyed Molybdenum at 1800 F, Room Temperature, and −40 F—A. A. Mittenbergs, D. N. Williams, and G. D. Haley, presented by Mr. Mittenbergs.

High-Temperature Fatigue and Bending Strain Measurements in Controlled Environments—G. J. Danek, H. H. Smith, and M. R. Achter, presented by Mr. Danek.

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[8] Published in *Materials Research & Standards*, Vol. 1, No. 9, p. 711, Sept., 1961.

ELEVENTH SESSION—SYMPOSIUM ON RADIATION EFFECTS IN REFRACTORY FUEL COMPOUNDS[9]

TUESDAY, JUNE 27, 9:30 A.M.

SESSION CHAIRMAN: BRUCE DUNNINGTON

Some Consequences of Excess Oxygen in UO_2—J. A. L. Robertson, presented by the author.

Voids in Irradiated UO_2—J. L. Bates, presented by the author.

The Heat Rating Required to Produce Central Melting in Various UO_2 Fuels—A. S. Bain, presented by the author.

Fission Fragment Tracks in UO_2—T. S. Noggle and J. O. Stiegler, presented by Mr. Noggle.

Effects of Burnup on Certain Ceramic Fuel Materials—M. L. Bleiberg, W. Yeniscavich, and R. G. Gray, presented by Mr. Bleiberg.

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[9] Issued as *ASTM STP No. 306*.

(Continued in Fourteenth, Nineteenth, and Twenty-first Sessions)

TWELFTH SESSION—PRESIDENT'S LUNCHEON AND BUSINESS MEETING

TUESDAY, JUNE 27, 12:00 NOON

SESSION CHAIRMAN: PAST-PRESIDENT F. L. LAQUE

There were 238 present at the luncheon session, including a number of ladies and visitors.

Election of Officers:

The results of the letter ballot on election of new officers were reported by D. I. Finch on behalf of the Teller's Committee consisting of Mr. Finch and Dr. G. D. Patterson, Jr. The results were as follows:

For President, to serve for one year:
  Miles N. Clair, 2742 votes.
For Vice-President, to serve for two years:
  Alfred C. Webber, 2725 votes.
For Directors, to serve for three years:
  Ardrey M. Bounds, 2685 votes.
  Albert G. H. Dietz, 2719 votes.
  Bruce W. Gonser, 2690 votes.

Gordon M. Kline, 2685 votes.
Wayne A. Kirklin, 2695 votes.
James B. Rather, Jr., 2665 votes.

The newly elected directors were introduced with the exception of Mr. Kirklin who was not present. President-elect Miles N. Clair and Vice-President-elect A. C. Webber were introduced in turn, and both responded briefly.

Recognition of Retiring Directors and Past-President:

President Bates presented certificates to those retiring from the Board of Directors. Mention was made that this meeting brought to a close for Richard T. Kropf nine years on the Board of Directors. Retiring directors present were W. L. Fink, H. M. Hancock and Richard T. Kropf. Unable to attend the luncheon were P. A. Archibald, L. A. O'Leary, and R. D. Thompson.

Award of Honorary Memberships:

The Executive Secretary read citations for the five members elected to Honorary Membership by action of the Board of Directors on May 9, 1961. The President then presented certificates of Honorary Membership to Edward J. Albert, Simon Collier, Stanton Walker, and W. A. Zinzow. The fifth Honorary Member, Theodore Parker Dresser, Jr., was unable to be present.

Special Award:

President A. Allan Bates, on behalf of the Board of Directors, presented a scroll "With sincere appreciation and in recognition of the distinguished service" to Robert J. Painter and conferred on him the title of "Executive Secretary Emeritus."

Presidential Address:

The annual President's address was presented by A. Allan Bates on the subject, "An ASTM for the Future.[10]"

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[10] See p. 46.

BUSINESS MEETING OF THE SOCIETY

Report of the Board of Directors:

T. A. Marshall, Jr., Executive Secretary, presented a résumé of the Annual Report of the Board of Directors.

Amendments of the Charter:

The Executive Secretary reported the adoption of the following resolutions by the Board of Directors at its meeting May 9, 1961:

RESOLVED that the Board of Directors of the AMERICAN SOCIETY FOR TESTING MATERIALS hereby approves as proposed amendments of the Charter of the Society (*a*) by amendment of Section 1 and (*b*) addition of a new Section 9, such amendments to read as follows:

1. The name of the Corporation is the "AMERICAN SOCIETY FOR TESTING AND MATERIALS."

9. Notwithstanding any future construction of any of the foregoing provisions to the contrary, the Corporation shall not engage in any activity which is not educational, technical, scientific, or charitable within the meaning of Section 501(c)(3) of the Internal Revenue Code, 1954, or equivalent provisions of any amendment of such Code, or of a superseding Internal Revenue Code, or other Federal law of like import, and on dissolution of the Corporation its assets shall be distributed to an organization or organizations organized and operated for similar educational, technical, scientific, or charitable purposes.

AND FURTHER RESOLVED that the Executive Secretary is directed forthwith to notify all members of the Society of the proposed amendments of the Charter and the reasons therefor, that consideration of, and action on the proposed amendments by the membership will take place at a meeting of the members to be held at 1:30 p.m., Tuesday, June 27, 1961, in the Carolina Room, Chalfonte Hotel, North Carolina Avenue and the Boardwalk, Atlantic City, New Jersey; that in such notification provision be made for members who are unable to attend the meeting to permit them to cast their vote for or against the proposed amendments, with the same force and effect as if cast in person by such members at such meeting; and finally, that if the action of the membership be favorable to such amendments the President and Executive Secretary are authorized and directed to present to the appropriate Court pertinent Articles of Amendment of the Charter, and to take such further action as may be required by the laws of the Commonwealth of Pennsylvania to effectuate said amendments.

He then reported the results of ballots received from members who were unable to attend the luncheon as follows: for adoption of the resolution to change the name of the Society, 3339; against adoption, 235; for adoption of the resolution to add Section 9, dissolution clause, to the Charter, 3506; against adoption, 16. After discussion from the floor, the 127 members present voted 126 to 1 to approve the proposed amendment to change the name of the Society. Unanimous approval was given to the proposal to add the dissolution clause to the Charter. The members voted also unanimous approval of the resolution set forth below:

RESOLVED that the AMERICAN SOCIETY FOR TESTING MATERIALS, in meeting assembled, after due and legal notice, in accordance with the laws of the Commonwealth of Pennsylvania, hereby approves the foregoing proposed amendments of the Charter of the Society by amendment of Section 1 and addition of a new Section 9, as set forth in the Resolutions adopted by the Board of Directors at its meeting held May 9, 1961, and the President and Executive Secretary are authorized and directed to present to the appropriate Court pertinent Articles of Amendment of the Charter, and to take such further action as may be required by the laws of the Commonwealth of Pennsylvania to effectuate said amendments.

Amendments of the By-laws:

The Executive Secretary reported the following proposed amendments of the Society's By-laws:

The Board of Directors at its meeting on January 17, 1961, received and approved for submission to the membership a petition signed by twelve members to amend the By-laws by amending Section 5 of Article VIII. Dues, by deletion of the words in parentheses:

> Sec. 5. Any person elected after six months of any fiscal year shall have expired, may pay only one-half of the amount of dues for that fiscal year; (but in that case he shall not be entitled to a copy of the Proceedings for the current year).

The Board recommended approval of this proposed amendment for submission to the membership by letter ballot due to revision in the system of distribution of the Proceedings.

At its meeting on May 9, 1961, the Board received and approved for submission to the membership a petition to amend the By-laws by amending Article IV. Duties of Officers, by renumbering Section 5 as Section 6 and inserting a new Section 5 to read as follows:

> Sec. 5. The Board of Directors may, in its discretion, appoint a member or members, or other person or persons, to represent it at meetings of societies of kindred aim or at public functions. Such delegates may be designated as "Honorary Vice-President," and their duties shall terminate with the occasion for which they are appointed.

And by amending Article VIII. Dues, by adding a new Section 6 to read as follows:

> Sec. 6. At its discretion, the Board of Directors may confer life membership upon any person who is an individual member.

The Board recommended approval of these amendments for submission to the membership by letter ballot.

Each amendment was voted upon separately, and the members present approved the submission of each amendment to letter ballot of the Society.

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Passing the Gavel:

Following the business meeting, President Bates presented the gavel of the president to President-elect Clair who in turn bestowed the Past-President's pin and certificate of service on President Bates.

THIRTEENTH SESSION—SYMPOSIUM ON EVALUATION OF METALLIC MATERIALS IN DESIGN FOR LOW-TEMPERATURE SERVICE[7]

(*Continued from the Ninth Session*)

TUESDAY, JUNE 27, 2:00 P.M.

SESSION CHAIRMAN: F. B. FOLEY
CO-CHAIRMAN: G. R. IRWIN

Steels of Improved Fracture Toughness—J. M. Hodge, presented by the author.

Testing Techniques and Evaluation of Materials for Use at Liquid Hydrogen Temperatures—R. Markovich and F. Schwartzberg, presented by the authors.

The Correlation of Notch:Unnotch Tensile Ratios with Tensile Fatigue Properties of Complex Welded Joints in High-Strength 300 Series Stainless Steels at Cryogenic Temperatures—J. F. Watson, J. L. Christian, T. T. Tanalski, and A. Hurlich, presented by Mr. Watson.

Some Factors Influencing the Fracture Toughness of Sheet Alloys for Use in Lightweight Cryogenic Tankage—G. B. Espey, M. H. Jones, and W. F. Brown, Jr., presented by Mr. Brown.

Design of Cryogenic Storage for Industrial Applications—H. W. Marsh, presented by the author.

Brittle-Fracture Transition of Some Concrete Reinforcing Steels—A. L. Tarr, presented by the author.

(*Continued in Twenty-second Session*)

FOURTEENTH SESSION—SYMPOSIUM ON RADIATION EFFECTS IN REFRACTORY FUEL COMPOUNDS[9]

(*Continued from the Eleventh Session*)

TUESDAY, JUNE 27, 2:30 P.M.

SESSION CHAIRMAN: J. B. TRICE

Study of the Factors Controlling the Release of Xe^{133} from Bulk UO_2—D. F. Toner and J. L. Scott, presented by Mr. Toner.

In-Pile Release of Fission Products in UO_2—J. B. Melehan and F. A. Rough, presented by Mr. Melehan.

The Continuous Release of Fission Gas from UO_2 During Irradiation—R. M. Carroll, presented by the author.

(*Continued in Nineteenth and Twenty-first Sessions*)

FIFTEENTH SESSION—COMMITTEE REPORT SESSION

TUESDAY, JUNE 27, 4:30 P.M.

SESSION CHAIRMAN: G. H. HARNDEN

Committee A-7 on Malleable-Iron Castings:

Report presented in the absence of the chairman by J. H. Lansing, secretary, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Pearlitic Malleable Iron Castings (A 220 - 55 T)

Adopted as Standard, Revisions in:

Spec. for Malleable Iron Castings (A 47 - 52)

Spec. for Malleable Iron Flanges, Pipe Fittings, and Valves and Parts for Railroad, Marine, and Other Heavy Duty Service (A 338 - 54)

Committee B-4 on Metallic Materials for Thermostats and for Electrical Resistance, Heating and Contacts:

Report presented in the absence of the chairman by C. K. Strobel, secretary, and the following actions taken:

Accepted for Publication as Information Only:

Proposed Def. of Terms Relating to Electric Contacts and Their Use

Adopted as Standard:

Test for Surety of Make of Electrical Contact Materials (B 340 - 59 T)

Test for Resistance Characteristics of Microcontacts (B 326 - 58 T)

Committee B-6 on Die-Cast Metals and Alloys:

Report presented by W. Babington, chairman, and accepted as a report of progress.

Committee C-4 on Clay Pipe:

Report presented by C. R. Velzy, chairman, and accepted as a report of progress.

Committee C-24 on Joint Sealants:

Report presented in the absence of the chairman by J. R. Panek, secretary, and accepted as a report of progress.

Committee D-24 on Carbon Black:

Report presented by N. P. Bekema, chairman, and the following actions taken:

Accepted as Tentative:

Method for Sampling Bulk Shipments of Carbon Black (D 1900 - 61 T), with the deletion of sentences 2 and 3 of Section 2, and editorial changes in Sections 1 and 3.

Accepted as Tentative, Revisions in:

Spec. for Carbon Blacks Used in Rubber Products (D 1765 - 60 T) (Jointly with Committee D-11)

Committee E-4 on Metallography:

Report presented by L. L. Wyman, chairman, and the following action taken:

Adopted as Standard:

Methods for Estimating the Average Grain Size of Metals (E 112 - 60 T)

Committee E-11 on Quality Control of Materials:

Report presented by Simon Collier, chairman, and the following actions taken:

Accepted as Tentative:

Rec. Practice for Use of the Terms Precision and Accuracy as Applied to Measurement of a Property of a Material (E 177 - 61 T)

Rec. Practice for Dealing with Outlying Observations (E 178 - 61 T)

Adopted as Standard:

Rec. Practice for the Acceptance of Evidence Based on the Results of Probability Sampling (E 141 - 59 T)

Committee F-1 on Materials for Electron Tubes and Semiconductor Devices:

Report presented in the absence of the chairman by C. Guettel, secretary, and the following actions taken subject to a favorable letter ballot vote in Committee F-1 on the report:

Accepted as Tentative:

Rec. Practice for Making and Testing Reference Glass-Metal Bead-Seal (F 14 - 61 T)

Specs. for Iron-Nickel-Cobalt Sealing Alloy (F 15 - 61 T)

Methods for Measuring Diameter or Thickness of Wire and Ribbon for Electronic Devices and Lamps (F 16 - 61 T)

Accepted as Tentative, Revisions in:

Spec. for Aluminum Oxide Powder (F 7 - 58 T)

Spec. for Nickel Alloy Cathode Sleeves for Electronic Devices (F 239 - 57 T)

Methods of Testing Sleeves and Tubing for Radio Tube Cathodes (F 128 - 56 T)

Rec. Practice for Cathode Melt Prove-In Testing (F 238 - 55 T)

SIXTEENTH SESSION—GILLETT MEMORIAL LECTURE[11]

TUESDAY, JUNE 27, 5:00 P.M.

SESSION CHAIRMAN: PRESIDENT A. ALLAN BATES

Tenth Gillett Memorial Lecture:

President Bates opened the session by giving a brief statement of the purpose of the lecture. This lecture is sponsored jointly by the ASTM and the Battelle Memorial Institute to perpetuate the memory of Horace W. Gillett, one of America's leading technologists, the first

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[11] Issued as a separate publication.

Gordon M. Kline, 2685 votes.
Wayne A. Kirklin, 2695 votes.
James B. Rather, Jr., 2665 votes.

The newly elected directors were introduced with the exception of Mr. Kirklin who was not present. President-elect Miles N. Clair and Vice-President-elect A. C. Webber were introduced in turn, and both responded briefly.

Recognition of Retiring Directors and Past-President:

President Bates presented certificates to those retiring from the Board of Directors. Mention was made that this meeting brought to a close for Richard T. Kropf nine years on the Board of Directors. Retiring directors present were W. L. Fink, H. M. Hancock and Richard T. Kropf. Unable to attend the luncheon were P. A. Archibald, L. A. O'Leary, and R. D. Thompson.

Award of Honorary Memberships:

The Executive Secretary read citations for the five members elected to Honorary Membership by action of the Board of Directors on May 9, 1961. The President then presented certificates of Honorary Membership to Edward J. Albert, Simon Collier, Stanton Walker, and W. A. Zinzow. The fifth Honorary Member, Theodore Parker Dresser, Jr., was unable to be present.

Special Award:

President A. Allan Bates, on behalf of the Board of Directors, presented a scroll "With sincere appreciation and in recognition of the distinguished service" to Robert J. Painter and conferred on him the title of "Executive Secretary Emeritus."

Presidential Address:

The annual President's address was presented by A. Allan Bates on the subject, "An ASTM for the Future.[10]"

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[10] See p. 46.

BUSINESS MEETING OF THE SOCIETY

Report of the Board of Directors:

T. A. Marshall, Jr., Executive Secretary, presented a résumé of the Annual Report of the Board of Directors.

Amendments of the Charter:

The Executive Secretary reported the adoption of the following resolutions by the Board of Directors at its meeting May 9, 1961:

RESOLVED that the Board of Directors of the AMERICAN SOCIETY FOR TESTING MATERIALS hereby approves as proposed amendments of the Charter of the Society (*a*) by amendment of Section 1 and (*b*) addition of a new Section 9, such amendments to read as follows:

1. The name of the Corporation is the "AMERICAN SOCIETY FOR TESTING AND MATERIALS."
9. Notwithstanding any future construction of any of the foregoing provisions to the contrary, the Corporation shall not engage in any activity which is not educational, technical, scientific, or charitable within the meaning of Section 501(c)(3) of the Internal Revenue Code, 1954, or equivalent provisions of any amendment of such Code, or of a superseding Internal Revenue Code, or other Federal law of like import, and on dissolution of the Corporation its assets shall be distributed to an organization or organizations organized and operated for similar educational, technical, scientific, or charitable purposes.

AND FURTHER RESOLVED that the Executive Secretary is directed forthwith to notify all members of the Society of the proposed amendments of the Charter and the reasons therefor, that consideration of, and action on the proposed amendments by the membership will take place at a meeting of the members to be held at 1:30 p.m., Tuesday, June 27, 1961, in the Carolina Room, Chalfonte Hotel, North Carolina Avenue and the Boardwalk, Atlantic City, New Jersey; that in such notification provision be made for members who are unable to attend the meeting to permit them to cast their vote for or against the proposed amendments, with the same force and effect as if cast in person by such members at such meeting; and finally, that if the action of the membership be favorable to such amendments the President and Executive Secretary are authorized and directed to present to the appropriate Court pertinent Articles of Amendment of the Charter, and to take such further action as may be required by the laws of the Commonwealth of Pennsylvania to effectuate said amendments.

He then reported the results of ballots received from members who were unable to attend the luncheon as follows: for adoption of the resolution to change the name of the Society, 3339; against adoption, 235; for adoption of the resolution to add Section 9, dissolution clause, to the Charter, 3506; against adoption, 16. After discussion from the floor, the 127 members present voted 126 to 1 to approve the proposed amendment to change the name of the Society. Unanimous approval was given to the proposal to add the dissolution clause to the Charter. The members voted also unanimous approval of the resolution set forth below:

RESOLVED that the AMERICAN SOCIETY FOR TESTING MATERIALS, in meeting assembled, after due and legal notice, in accordance with the laws of the Commonwealth of Pennsylvania, hereby approves the foregoing proposed amendments of the Charter of the Society by amendment of Section 1 and addition of a new Section 9, as set forth in the Resolutions adopted by the Board of Directors at its meeting held May 9, 1961, and the President and Executive Secretary are authorized and directed to present to the appropriate Court pertinent Articles of Amendment of the Charter, and to take such further action as may be required by the laws of the Commonwealth of Pennsylvania to effectuate said amendments.

Amendments of the By-laws:

The Executive Secretary reported the following proposed amendments of the Society's By-laws:

The Board of Directors at its meeting on January 17, 1961, received and approved for submission to the membership a petition signed by twelve members to amend the By-laws by amending Section 5 of Article VIII. Dues, by deletion of the words in parentheses:

> Sec. 5. Any person elected after six months of any fiscal year shall have expired, may pay only one-half of the amount of dues for that fiscal year; (but in that case he shall not be entitled to a copy of the Proceedings for the current year).

The Board recommended approval of this proposed amendment for submission to the membership by letter ballot due to revision in the system of distribution of the Proceedings.

At its meeting on May 9, 1961, the Board received and approved for submission to the membership a petition to amend the By-laws by amending Article IV. Duties of Officers, by renumbering Section 5 as Section 6 and inserting a new Section 5 to read as follows:

> Sec. 5. The Board of Directors may, in its discretion, appoint a member or members, or other person or persons, to represent it at meetings of societies of kindred aim or at public functions. Such delegates may be designated as "Honorary Vice-President," and their duties shall terminate with the occasion for which they are appointed.

And by amending Article VIII. Dues, by adding a new Section 6 to read as follows:

> Sec. 6. At its discretion, the Board of Directors may confer life membership upon any person who is an individual member.

The Board recommended approval of these amendments for submission to the membership by letter ballot.

Each amendment was voted upon separately, and the members present approved the submission of each amendment to letter ballot of the Society.

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Passing the Gavel:

Following the business meeting, President Bates presented the gavel of the president to President-elect Clair who in turn bestowed the Past-President's pin and certificate of service on President Bates.

THIRTEENTH SESSION—SYMPOSIUM ON EVALUATION OF METALLIC MATERIALS IN DESIGN FOR LOW-TEMPERATURE SERVICE[7]

(*Continued from the Ninth Session*)

TUESDAY, JUNE 27, 2:00 P.M.

SESSION CHAIRMAN: F. B. FOLEY
CO-CHAIRMAN: G. R. IRWIN

Steels of Improved Fracture Toughness—J. M. Hodge, presented by the author.

Testing Techniques and Evaluation of Materials for Use at Liquid Hydrogen Temperatures—R. Markovich and F. Schwartzberg, presented by the authors.

The Correlation of Notch:Unnotch Tensile Ratios with Tensile Fatigue Properties of Complex Welded Joints in High-Strength 300 Series Stainless Steels at Cryogenic Temperatures—J. F. Watson, J. L. Christian, T. T. Tanalski, and A. Hurlich, presented by Mr. Watson.

Some Factors Influencing the Fracture Toughness of Sheet Alloys for Use in Lightweight Cryogenic Tankage—G. B. Espey, M. H. Jones, and W. F. Brown, Jr., presented by Mr. Brown.

Design of Cryogenic Storage for Industrial Applications—H. W. Marsh, presented by the author.

Brittle-Fracture Transition of Some Concrete Reinforcing Steels—A. L. Tarr, presented by the author.

(*Continued in Twenty-second Session*)

FOURTEENTH SESSION—SYMPOSIUM ON RADIATION EFFECTS IN REFRACTORY FUEL COMPOUNDS[9]

(*Continued from the Eleventh Session*)

TUESDAY, JUNE 27, 2:30 P.M.

SESSION CHAIRMAN: J. B. TRICE

Study of the Factors Controlling the Release of Xe^{133} from Bulk UO_2—D. F. Toner and J. L. Scott, presented by Mr. Toner.

In-Pile Release of Fission Products in UO_2—J. B. Melehan and F. A. Rough, presented by Mr. Melehan.

The Continuous Release of Fission Gas from UO_2 During Irradiation—R. M. Carroll, presented by the author.

(*Continued in Nineteenth and Twenty-first Sessions*)

FIFTEENTH SESSION—COMMITTEE REPORT SESSION

TUESDAY, JUNE 27, 4:30 P.M.

SESSION CHAIRMAN: G. H. HARNDEN

Committee A-7 on Malleable-Iron Castings:

Report presented in the absence of the chairman by J. H. Lansing, secretary, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Pearlitic Malleable Iron Castings (A 220 - 55 T)

Adopted as Standard, Revisions in:

Spec. for Malleable Iron Castings (A 47 - 52)

Spec. for Malleable Iron Flanges, Pipe Fittings, and Valves and Parts for Railroad, Marine, and Other Heavy Duty Service (A 338 - 54)

Committee B-4 on Metallic Materials for Thermostats and for Electrical Resistance, Heating and Contacts:

Report presented in the absence of the chairman by C. K. Strobel, secretary, and the following actions taken:

Accepted for Publication as Information Only:

Proposed Def. of Terms Relating to Electric Contacts and Their Use

Adopted as Standard:

Test for Surety of Make of Electrical Contact Materials (B 340 - 59 T)

Test for Resistance Characteristics of Microcontacts (B 326 - 58 T)

Committee B-6 on Die-Cast Metals and Alloys:

Report presented by W. Babington, chairman, and accepted as a report of progress.

Committee C-4 on Clay Pipe:

Report presented by C. R. Velzy, chairman, and accepted as a report of progress.

Committee C-24 on Joint Sealants:

Report presented in the absence of the chairman by J. R. Panek, secretary, and accepted as a report of progress.

Committee D-24 on Carbon Black:

Report presented by N. P. Bekema, chairman, and the following actions taken:

Accepted as Tentative:

Method for Sampling Bulk Shipments of Carbon Black (D 1900 - 61 T), with the deletion of sentences 2 and 3 of Section 2, and editorial changes in Sections 1 and 3.

Accepted as Tentative, Revisions in:

Spec. for Carbon Blacks Used in Rubber Products (D 1765 - 60 T) (Jointly with Committee D-11)

Committee E-4 on Metallography:

Report presented by L. L. Wyman, chairman, and the following action taken:

Adopted as Standard:

Methods for Estimating the Average Grain Size of Metals (E 112 - 60 T)

Committee E-11 on Quality Control of Materials:

Report presented by Simon Collier, chairman, and the following actions taken:

Accepted as Tentative:

Rec. Practice for Use of the Terms Precision and Accuracy as Applied to Measurement of a Property of a Material (E 177 - 61 T)

Rec. Practice for Dealing with Outlying Observations (E 178 - 61 T)

Adopted as Standard:

Rec. Practice for the Acceptance of Evidence Based on the Results of Probability Sampling (E 141 - 59 T)

Committee F-1 on Materials for Electron Tubes and Semiconductor Devices:

Report presented in the absence of the chairman by C. Guettel, secretary, and the following actions taken subject to a favorable letter ballot vote in Committee F-1 on the report:

Accepted as Tentative:

Rec. Practice for Making and Testing Reference Glass-Metal Bead-Seal (F 14 - 61 T)

Specs. for Iron-Nickel-Cobalt Sealing Alloy (F 15 - 61 T)

Methods for Measuring Diameter or Thickness of Wire and Ribbon for Electronic Devices and Lamps (F 16 - 61 T)

Accepted as Tentative, Revisions in:

Spec. for Aluminum Oxide Powder (F 7 - 58 T)

Spec. for Nickel Alloy Cathode Sleeves for Electronic Devices (F 239 - 57 T)

Methods of Testing Sleeves and Tubing for Radio Tube Cathodes (F 128 - 56 T)

Rec. Practice for Cathode Melt Prove-In Testing (F 238 - 55 T)

SIXTEENTH SESSION—GILLETT MEMORIAL LECTURE[11]

TUESDAY, JUNE 27, 5:00 P.M.

SESSION CHAIRMAN: PRESIDENT A. ALLAN BATES

Tenth Gillett Memorial Lecture:

President Bates opened the session by giving a brief statement of the purpose of the lecture. This lecture is sponsored jointly by the ASTM and the Battelle Memorial Institute to perpetuate the memory of Horace W. Gillett, one of America's leading technologists, the first

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[11] Issued as a separate publication.

Director of the Institute, and for many years a very active worker in the Society, through the presentation of a lecture on a subject pertaining to the development, testing, evaluation, and application of metals.

President Bates called upon B. D. Thomas, President, Battelle Memorial Institute, who introduced Augustus B. Kinzel, Vice-President, Research, Union Carbide Corp. Mr. Kinzel presented the tenth H. W. Gillett Memorial Lecture on the subject "Specifications?!." The title of the lecture refers to the why, how, and finality of specifications. It included a brief discussion of the nature, history, purpose, and use of specifications. Topics explored included: the structure and content of specifications in relation to their objectives; the need for flexibility and judgment as a substitute for ignorance; and the relation of tests and testing to specifications, together with the effect of the nature of the test on the nature of the specification.

The possibilities of specifying in such a way as to make it easier to consider the use of materials beyond their elastic limit were set forth. The role of engineering experience in specifications and approaches to the true technical worth of a material was examined. Finally, as part of the story of specifications and engineering, the need for engineering research and its place in today's picture were stressed with the thought that specifications may be provided that do have real meaning and finality with respect to specific applications.

President Bates expressed appreciation to Mr. Kinzel for his outstanding lecture and presented to him on behalf of the Society the H. W. Gillett Memorial Lecture Certificate.

SEVENTEENTH SESSION—SESSION ON HIGH TEMPERATURE

TUESDAY, JUNE 27, 8:00 P.M.

SESSION CHAIRMAN: J. J. KANTER

Apparatus for Obtaining Mechanical Properties at High Temperatures—D. H. Fisher, R. L. Carlson, and F. C. Holden, presented by Mr. Fisher.[12]

Elevated Temperature Dynamic Moduli of Metallic Materials—W. H. Hill, K. D. Shimmin, and B. A. Wilcox, presented by Mr. Shimmin.

The Applications of Rabotnov's Creep Parameter—R. M. Goldhoff, presented by the author.

Determination of Compressive, Bearing, and Shear Creep of Sheet Metals—J. R. Kattus and H. L. Lessley, presented by Mr. Kattus.

Creep of Structural Joints from Simple Tension Creep—Joseph Marin, presented by the author.

(*Continued in Twenty-sixth Session*)

EIGHTEENTH SESSION—SESSION ON CONCRETE

(*Continued from the Eighth Session*)

TUESDAY, JUNE 27, 8:00 P.M.

SESSION CHAIRMAN: STANTON WALKER

Glue! For Concrete Repair?—C. M. Wakeman, H. E. Stover, and E. N. Blye, presented by M. R. Smith.[13]

Variation on Pozzolanic Behavior of Fly Ashes—R. D. Vincent, Manuel Mateos, and D. T. Davidson, presented by Mr. Mateos.

Concrete Containing Fly Ash as a Replacement for Portland Blast-Furnace Slag Cement—W.

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[12] Published in *Materials Research & Standards*, Vol. 2, No. 1, Jan., 1962.

[13] Published in *Materials Research & Standards*, Vol. 2, No. 2, Feb., 1962.

E. Grieb and D. O. Woolf, presented by Mr. Grieb.

Testing Uniformity of Large Batches of Concrete—D. L. Bloem, R. D. Gaynor, and J. R. Wilson, presented by Mr. Bloem.

Modern Concepts in Control of Concrete—J. J. Waddell, presented by the author.[14]

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[14] Published in *Materials Research & Standards*, Vol. 1, No. 6, p. 452, June 1961.

NINETEENTH SESSION—SYMPOSIUM ON RADIATION EFFECTS IN REFRACTORY FUEL COMPOUNDS[9]

(Continued from Eleventh and Fourteenth Sessions)

TUESDAY, JUNE 27, 8:00 P.M.

SESSION CHAIRMAN: M. J. WHITMAN

Panel Discussion on Irradiation Test Methods:

Panel Members: J. A. Brinkman
D. S. Billington
J. A. L. Robertson
J. L. Bates
E. A. Aitken
M. L. Bleiberg

(Continued in Twenty-first Session)

TWENTIETH SESSION—SYMPOSIUM ON EXTENSION OF SENSITIVITY FOR DETERMINING VARIOUS CONSTITUENTS IN METALS[15]

WEDNESDAY, JUNE 28, 9:00 A.M.

SESSION CHAIRMAN: L. M. MELNICK

Separations in Analysis—J. L. Hague, presented by the author.

Recent Developments in Electroanalytical Chemistry—Louis Meites, presented by the author.

Radioactivation Analysis: A Sensitive and Specific Method for Metals Analysis—G. W. Leddicotte, presented by the author.

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[15] Issued as *ASTM STP No. 308.*

(Continued in Twenty-fifth Session)

TWENTY-FIRST SESSION—SYMPOSIUM ON RADIATION EFFECTS IN REFRACTORY FUEL COMPOUNDS[9]

(Continued from the Eleventh, Fourteenth and Nineteenth Sessions)

WEDNESDAY, JUNE 28, 9:30 A.M.

SESSION CHAIRMAN: J. E. GATES

Sintering Characteristics in a Radiation Environment—E. A. Aitken, presented by the author.

Mechanism of Irradiation Damage in Uranium Monocarbide—D. G. Freas, A. E. Austin, and F. A. Rough, presented by D. G. Freas.

Hydrolysis of Beryllia—C. C. Browne, presented by the author.

The Irradiation and Examination of a PuO_2-UO_2 Fast Reactor Oxide Fuel—J. N. Siltanen, J. M. Gerhart, and J. S. Cochran, presented by Mr. Gerhart.

TWENTY-SECOND SESSION—SYMPOSIUM ON EVALUATION OF METALLIC MATERIALS IN DESIGN FOR LOW-TEMPERATURE SERVICE[7]

(Continued from the Ninth and Thirteenth Sessions)

WEDNESDAY, JUNE 28, 9:00 A.M.

SESSION CHAIRMAN: G. R. IRWIN
CO-CHAIRMAN: F. B. FOLEY

Testing and Design Considerations in Brittle Fracture—Sumio Yukawa, presented by the author.

Influence of Sheet Thickness on the Sharp Edge Notch Properties of a β Titanium Alloy at Room and Low Temperatures—A. J. Repko, M. H. Jones, and W. F. Brown, Jr., presented by title only.

Panel Discussion:

Panel Members: Authors
G. R. Irwin, U. S. Naval Research Laboratory.
Robert Stout, Lehigh University.
P. P. Puzak, U. S. Naval Research Laboratory.
John Low, Jr., General Electric Co.

TWENTY-THIRD SESSION—COMMITTEE REPORT SESSION

WEDNESDAY, JUNE 28, 11:30 A.M.

SESSION CHAIRMAN: VICE-PRESIDENT MILES N. CLAIR

Committee C-8 on Refractories:

Report presented in the absence of the chairman by S. F. Etris, ASTM Staff, and the following actions taken:

Accepted as Tentative:

Classification of Mullite Refractories (C 467 - 61 T)
Classification of Refractory Granular Dolomite (C 468 - 61 T)

Accepted as Tentative, Revisions in:

Test for Modulus of Rupture of Castable Refractories (C 268 - 55 T)
Test for Permanent Linear Change on Firing of Castable Refractories (C 269 - 57 T)
Classification of Steel Pouring Pit Refractories (C 435 - 59 T)
Classification of Fireclay and High-Alumina Refractory Brick (C 27 - 60)
Method for Basic Procedure in Panel Spalling Test for Refractory Brick (C 38 - 58)
Def. of Terms Relating to Refractories (C 71 - 55) (Revision of existing tentative revision and the addition of a new tentative revision)
Method of Panel Spalling Test for High Duty Fireclay Brick (C 107 - 52)
Method of Panel Spalling Test for Super Duty Fireclay Brick (C 122 - 52)
Classification of Insulating Fire Brick (C 155-57)
Method of Panel Spalling Test for Fireclay Plastic Refractories (C 180 - 52)
Spec. for Refractories for Incinerators (C 106 - 51) (Revision of existing tentative revision)
Specs. for Fireclay-Base Castable Refractories for Boiler Furnaces and Incinerators (C 213 - 58) (Standard reverted to tentative)

Adopted as Standard:

Test for Reheat Change of Carbon Brick and Shapes (C 436 - 59 T)
Test for Size and Bulk Density of Insulating Fire Brick (C 437 - 59 T)
Test for Thermal Conductivity of Plastic Refractories (C 438 - 59 T)
Test for Resistance to Thermal Spalling of Silica Brick (C 439 - 59 T)
Spec. for Insulating Fire Brick for Linings of Industrial Furnaces Operated with a Neutral or Oxidizing Atmosphere (C 434 - 59 T)

Adopted as Standard, Revisions in:

Test for Reheat Change of Refractory Brick (C 113-46)
Spec. for Refractories for Malleable Iron Furnaces with Removable Bungs, and for Annealing Ovens (C 63 - 51)
Spec. for Refractories for Heavy Duty Stationary Boiler Service (C 64 - 51)
Spec. for Refractories for Moderate Duty Stationary Boiler Service (C 153-51)

Test for Reheat Change of Insulating Fire Brick (C 210 – 46)

Committee C-14 on Glass and Glass Products:

Report presented in the absence of the chairman by S. F. Etris, ASTM Staff, and the following action taken:

Adopted as Standard, Revisions in:

Methods of Testing Structural Non-Load-Bearing Cellular Glass Blocks (C 240 – 56)

Committee C-16 on Thermal Insulating Materials:

Report presented in the absence of the chairman by S. F. Etris, ASTM Staff, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Mineral Fiber Felt Insulation (Industrial Type) for Elevated Temperatures (C 382 – 56 T)
Def. of Terms Relating to Thermal Insulating Materials (C 168 – 56)
Methods of Sampling and Mixing Thermal Insulating Cement (C 163 – 44) (Standard reverted to tentative)

Adopted as Standard:

Spec. for Expanded or Exfoliated Vermiculite Thermal Insulating Cement (C 196 – 59 T)
Spec. for Diatomaceous Silica Thermal Insulating Cement (C 197 – 59 T)
Test for Mean Specific Heat of Thermal Insulation (C 351 – 59 T)
Test for Hot Surface Performance of High-Temperature Thermal Insulation (C 411 – 58 T)
Test for Weight Loss by Tumbling of Preformed Thermal Insulation (C 421 – 58 T)
Test for Normal Total Emittance of Surfaces of Materials 0.01 in. or Less in Thickness at Approximately Room Temperature (C 445 – 59 T)

Adopted as Standard, Revisions in:

Tests for Covering Capacity and Volume Change upon Drying of Thermal Insulating Cement (C 166 – 52)

Standard Withdrawn:

Test for Storage Density of Thermal Insulating Cement (C 164 – 52)

Committee C-18 on Natural Building Stones:

Report presented in the absence of the chairman by S. F. Etris, ASTM Staff, and accepted as a report of progress.

Committee C-19 on Structural Sandwich Constructions:

Report presented in the absence of the chairman by S. F. Etris, ASTM Staff, and the following actions taken:

Accepted as Tentative:

Test for Flexure-Creep of Sandwich Constructions (C 480 – 61 T)
Test for Laboratory Aging of Sandwich Constructions (C 481 – 6 T)

Accepted as Tentative, Revisions in:

Method of Flatwise Flexure Test of Sandwich Constructions (C 393 – 57 T)

Adopted as Standard, Revisions in:

Test for Density of Core Materials for Structural Sandwich Constructions (C 271 – 53)
Method of Shear Test in Flatwise Plane of Sandwich Constructions (C 273 – 53)
Method of Tension Test in Flatwise Plane of Sandwich Constructions (C 297 – 55)
Test for Edgewise Compressive Strength of Flat Sandwich Constructions (C 364 – 57)

Committee C-20 on Acoustical Materials:

Report presented in the absence of the chairman by Ralph Huntley, secretary, and accepted as a report of progress.

Committee C-21 on Ceramic Whitewares and Related Products:

Report presented in the absence of the chairman by S. F. Etris, ASTM Staff, and the following actions taken:

Accepted as Tentative:

Test for Thermal Shock Resistance of Glazed Ceramic Tile (C 484 – 61 T)
Method for Measuring Warpage of Ceramic Tile (C 485 – 61 T)
Test for Electrical Resistance of Conductive Ceramic Tile (C 483 – 61 T)
Test for Bond Strength of Glazed Interior Ceramic Wall Tile in Portland-Cement Installations (C 482 – 61 T)

Committee C-22 on Porcelain Enamel:

Report presented in the absence of the chairman by Michael Bozsin, and the following actions taken:

Accepted as Tentative:

Test for Spalling Resistance of Porcelain Enameled Aluminum (C 486 – 61 T)

Accepted as Tentative, Revisions in:

Def. of Terms Relating to Porcelain Enamel (C 286 – 58 T)

Tests for Acid Resistance of Porcelain Enamels (C 282 – 53) (Standard reverted to tentative)

Adopted as Standard:

Tests for Abrasion Resistance of Porcelain Enamels (C 448 – 59 T)

Committee D-22 on Methods of Atmospheric Sampling and Analysis:

Report presented in the absence of the chairman by S. F. Etris, ASTM Staff, and the following actions taken:

Accepted as Tentative:

Test for Mass Concentration of Particulate Matter in the Atmosphere (Continuous-Measurement Light-Scattering Method) (D 1899 – 61 T)

Adopted as Standard:

Test for Particulate Matter in the Atmosphere (Optical Density of Filtered Deposit) (D 1704 – 59 T)

Committee D-23 on Cellulose and Cellulose Derivatives:

Report presented in the absence of the chairman by S. F. Etris, ASTM Staff, and the following actions taken:

Accepted for Publication as Information Only:

Proposed Test for Carbonyl Content of Cellulose

Accepted as Tentative, Revisions in:

Methods of Testing Sodium Carboxylmethylcellulose (D 1439 – 58 T)

Methods of Testing Cellulose Acetate (D 871 – 56) (Standard reverted to tentative)

Adopted as Standard:

Tests for Moisture in Cellulose (D 1348 – 58 T)

Test for Solubility of Cellulose in Sodium Hydroxide (D 1696 – 59 T)

Committee E-3 on Chemical Analysis of Metals:

Report presented in the absence of the chairman by S. F. Etris, ASTM Staff, and accepted as a report of progress.

TWENTY-FOURTH SESSION—AWARDS LUNCHEON

WEDNESDAY, JUNE 28, 12:00 NOON

SESSION CHAIRMAN: PAST-PRESIDENT RICHARD T. KROPF

About 315 were present at this luncheon session, including a number of ladies and visitors.

Recognition of 40-Year, 50-Year, and 60-Year Members:

Past-President Kropf referred to the list of 15 personal and 36 company members who had maintained continuous membership in ASTM for 40 years and introduced those individual members and company representatives who were present.

He then introduced those 50-year members who were present, noting that there were 18 in the group, including three individual members.

This year, for the first time, recognition was given to those whose membership in the Society had been maintained for 60 years or more, and the chairman introduced the representatives of 11 company members in this category to the luncheon audience. There were no individual members in this category.

Certificates of membership were awarded to the 40-, 50-, and 60-year members and representatives at the close of the luncheon.

Guest Speaker:

Rear Admiral M. J. Lawrence, USN, Assistant Chief for Technical Logistics, Bureau of Ships, spoke on "Fifty Years of ASTM Membership.[16]"

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[16] Published in *Materials Research & Standards*, Vol. 1, No. 8, p. 641, Aug., 1961.

Announcements of Awards:

Past-President Kropf announced that the following awards had been conferred prior to the Awards Luncheon.

The Harold DeWitt Smith Award was given to Lyman E. Fourt, Assistant Director, Harris Research Laboratories, Washington, D. C., for outstanding achievement in research on fibers and their properties. It was presented at a meeting of Committee D-13 on Textile Materials on March 2, 1961.

The Max Hecht Award was presented to Claude K. Rice of Venezuela Gulf Refining Co. for his outstanding contributions to the work of Committee D-19 on Industrial Water at a special luncheon on April 22, 1961.

The C. A. Hogentogler Award was presented to C. B. Crawford, Division of Building Research, National Research Council of Canada, during the Seventh Session on Soils, Monday, June 26.

Fellowships and Grants-in-Aid:

Mr. Kropf introduced the chairman of the Committee on Fellowships and Grants-in-Aid, Past-President R. A. Schatzel, Vice-President, Rome Cable Division, Aluminum Company of America, who presented a brief report for the committee. The $6500 ASTM Doctoral Fellowship for the 1961–62 academic year was presented to the Massachusetts Institute of Technology. The five $1000 grants-in-aid were awarded as follows: Dr. J. Morrow, University of Illinois; Dr. Marvin A. Wilkov, The Pennsylvania State University; Dr. F. A. Hanes, Queens University (Ontario); Dr. John H. Koenig, Rutgers University; and Dr. Harold E. Way, Union College.

Dr. Schatzel announced that the Doctoral Fellowship for the 1962–63 academic year will be awarded to Purdue University. The recipients of the five grants-in-aid will be selected during the winter and announced during Committee Week.

Conferral of Awards and Medals:

Sanford E. Thompson Award.—The Sanford E. Thompson Award was given to Leonard Pepper and Bryant Mather, U. S. Army Engineer Waterways Experiment Station, for their paper, "Effectiveness of Mineral Admixtures in Preventing Excessive Expansion of Concrete Due to Alkali-Aggregate Reaction," published in the 1959 *Proceedings*. The recipients were introduced to President Bates by President-elect M. N. Clair, President, The Thompson & Lichtner Co.

Sam Tour Award.—The Sam Tour Award was presented to A. K. Graham and H. L. Pinkerton, of Graham, Savage and Associates, Inc., for their paper on "Standardizing the Preparation of Electrodeposits on Test Panels for Corrosion Testing," published in the 1959 *Proceedings*. The recipients were introduced by the Executive Secretary.

Richard L. Templin Award.—Prof. Wm. N. Findley, Brown University, chairman of the awards committee, announced the conferral of this award on E. A. Neppiras, Mullard Equipment Ltd., London, England, for his paper, "Techniques and Equipment for Fatigue Testing at Very High Frequencies," published in the 1959 *Proceedings*. Mr. Neppiras was unable to attend the luncheon.

Charles B. Dudley Medal.—The Charles B. Dudley Medal was conferred on G. B. Espey, M. H. Jones, and W. F. Brown, Jr., National Aeronautics and Space Administration, for their paper, "The Sharp Edge Notch Tensile Strength of Several High-Strength Steel Sheet Alloys," published in the 1959 *Proceedings*. The recipients were introduced by C. H. Adams, Monsanto Chemical Co., chairman of the awards committee.

Awards of Merit:

The chairman of the Award of Merit Committee, Dr. G. D. Patterson, Jr.,

E. I. du Pont de Nemours and Co., Inc., read brief citations and presented the following to President A. Allan Bates who conferred on them, on behalf of the Board of Directors, the Certificate of Award of Merit:

John J. Allen
Frank E. Clarke
Thomas E. Eagan
David F. Gould
Russell B. Gunia
Francis M. Howell
Richard S. Hunter
James H. Lansing
William Lerch
Robert R. Litehiser
Percy L. Rogers
Arthur G. Scroggie[a]
Hubert R. Snoke
John W. Whittemore
Lyman W. Wood
John S. Worth
Anthony M. Schwartz

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[a] J. A. Truitt of American Viscose Corp. received the award for Dr. Scroggie who was unable to be present.

TWENTY-FIFTH SESSION—SYMPOSIUM ON EXTENSION OF SENSITIVITY FOR DETERMINING VARIOUS CONSTITUENTS IN METALS [15]

(Continued from the Twentieth Session)

WEDNESDAY, JUNE 28, 2:00 P.M.

SESSION CHAIRMAN: N. E. GORDON, JR.

Ultratrace Emission Spectroscopy.—H. Morrison and R. L. Rupp, presented by Mr. Morrison.

Use of the Electron Probe to Measure Low Average but High Local Concentrations—L. S. Birks and R. E. Seebold, presented by Mr. Birks.

Extension of Sensitivity in the Analysis of Impurities in Solids by Mass Spectrometry—C. M. Stevens, presented by the author.

TWENTY-SIXTH SESSION—SESSION ON HIGH TEMPERATURE

(Continued from the Seventeenth Session)

WEDNESDAY, JUNE 28, 2:30 P.M.

SESSION CHAIRMAN: R. W. FENN

Papers:

The Creep and Rupture Properties of Five Copper-Base Casting Alloys—D. P. Moon and W. F. Simmons, presented by Mr. Moon.

Anomalous Fracture in the Creep of Nickel—T. C. Reuther, P. Shahinian, and M. R. Achter, presented by Mr. Reuther.

Creep-Rupture and Fatigue Properties of Three Nickel-Base Alloys in Helium Environment—F. J. Wall, H. B. Gayley, and T. F. Hengstenberg, presented by Mr. Wall.

Properties of Hardened Copper Beryllium Strip After Exposures to Elevated Temperatures—K. G. Wikle and N. P. Sarle, presented by Mr. Wikle.

Joint Committee on Effect of Temperature on the Properties of Metals:

Report presented by J. J. Kanter, chairman, and accepted as a report of progress.

TWENTY-SEVENTH SESSION—SESSION ON STEEL

WEDNESDAY, JUNE 28, 2:30 P.M.

SESSION CHAIRMAN: VICE-PRESIDENT R. W. SENIFF

A Specimen for Use in Investigating the Stress-Corrosion Cracking of Metals at Elevated Temperatures—H. L. Logan, presented by the author.[17]

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[17] Published in *Materials Research & Standards*, Vol. 2, No. 2, Feb., 1962.

A Fractrographic Analysis of 4340 Steel Missile Motor Casings—N. A. Tiner, presented by the author.

Strain Strengthening of Type 304 Stainless Steel—E. E. Weismantel, Walter Hammer, Jr. and L. G. Stemann, presented by Mr. Weismantel.

Effects of Variables in Charpy Impact Testing—N. H. Fahey, presented by the author.[18]

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[18] Published in *Materials Research & Standards*, Vol. 1, No. 11, p. 872, Nov., 1961.

DISCUSSION ON NON-COMMERCIAL MICROSCOPIC ACCESSORIES

WEDNESDAY, JUNE 28, 2:00 P.M.

SESSION CHAIRMAN: T. G. ROCHOW

SECRETARY AND CHAIRMAN OF DISCUSSION SESSION: M. C. MILLER

In accordance with the purposes of Subcommittee 28 on Microscopy, of Committee E-1 on Methods of Testing, to develop standards and methods applicable in industrial microscopy and to sponsor symposiums and collect and distribute microscopical information, this session on accessories was organized.

To stimulate discussion from the floor, the following several brief presentations were made:

Advantageous Use of Infinity-Corrected Objectives with Metals, Ceramics, Resins, etc., by Reflected Light—Peter Bartels.

A Specimen Stretcher and a Thermally-Controlled Stage for the Electron Microscope—Harvey B. Merrill, Jr.

Simple Physical Methods for Producing Microscopical Contrast—Sanford B. Newman.

These Were Not Worth Publishing, Four Orphans—Charles P. Saylor

Automatic Polisher—W. A. O. Olsen

PANEL DISCUSSION ON NUCLEAR STANDARDIZATION ACTIVITIES

WEDNESDAY, JUNE 28, 2:30 P.M.

This panel discussion was sponsored by the special ASTM Administrative Committee on Nuclear Problems to have presented the general scope of nuclear standardization activities in the American Society for Testing Materials, American Nuclear Society, The American Society of Mechanical Engineers, American Standards Assn., and Pressure Vessel Research Council. Also discussed were ways and means of coordinating current and future activities of the groups working on special projects in the various societies.

TWENTY-EIGHTH SESSION—COMMITTEE REPORT SESSION

WEDNESDAY, JUNE 28, 4:30 P.M.

SESSION CHAIRMAN: A. M. BOUNDS

Committee A-1 on Steel:

Report presented by J. J. Kanter, chairman, and the following actions taken:

Accepted as Tentative:

Method and Spec. for Magnetic Particle Inspection of Large Crankshaft Forgings (A 456 - 61 T)

Spec. for Centrifugally Cast Austenitic Steel Pipe for High-Temperature Service (A 451 - 61 T) (Jointly with Committee A-10)

Spec. for Centrifugally Cast Austenitic Cold-Wrought Pipe for High-Temperature Service (A 452 - 61 T) (Jointly with Committee A-10)

Spec. for High Tensile Strength Carbon-Manganese Steel Plates for Unfired Pressure Vessels (A 455 - 61 T)

Spec. for High-Strength, High-Temperature Bolting Materials with Expansion Coefficients

Comparable to Austenitic Steels (A 453 - 61 T) (Jointly with Committee A-10)

Spec. for Steel Conveyor Chain (A 454 - 61 T)

Accepted as Tentative, Revisions in:

Spec. for General Requirements for Delivery of Rolled Steel Plates, Shapes, Sheet Piling, and Bars for Structural Use (A 6 - 60 T)

Spec. for Steel for Bridges and Buildings (A 7 - 58 T)

Spec. for General Requirements for Hot-Rolled and Cold-Finished Carbon and Alloy Steel Bars (A 29 - 60 T)

Spec. for Structural Steel (A 36 - 60 T)

Spec. for Welded and Seamless Steel Pipe (A 53 - 60 T)

Specs. for Cold-Drawn Steel Wire for Concrete Reinforcement (A 82 - 58 T)

Specs. for Seamless Steel Boiler Tubes (A 83 - 60 T)

Specs. for Forged or Rolled Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 105 - 59 T), with a change in the new Section 4(*e*) to read as follows:

> (*e*) Forgings intended for application to pressure vessels may be repair welded provided the welding is performed in accordance with all the requirements of Part UF of Section VIII of the ASME Boiler and Pressure Vessel Code, and the repair welding is approved by the purchaser.

Specs. for Seamless Carbon-Steel Pipe for High-Temperature Service (A 106 - 60 T)

Spec. for Hot-Rolled Carbon Steel Bars (A 107 - 59 T)

Spec. for Cold-Finished Carbon Steel Bars and Shafting (A 108 - 58 T)

Specs. for Black and Hot-Dipped Zinc-Coated (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (A 120 - 57 T)

Specs. for Electric-Resistance-Welded Steel Pipe (A 135 - 60 T)

Spec. for Seamless Low-Carbon and Carbon-Molybdenum Steel Still Tubes for Refinery Service (A 161 - 60 T)

Specs. for Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 - 60 T)

Specs. for Seamless Cold-Drawn Low-Carbon Steel Heat-Exchanger and Condenser Tubes (A 179 - 60 T)

Specs. for Forged or Rolled Steel Pipe Flanges, Forged Fittings, and Valves and Parts for General Service (A 181 - 59 T), with a change in the new Section 4(*f*) to read as indicated above for the new Section 4(*e*) of Specifications A 105.

Specs. for Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 182 - 59 T)

Specs. for Welded Steel Wire Fabric for Concrete Reinforcement (A 185 - 58 T)

Specs. for Seamless Steel Boiler Tubes for High-Pressure Service (A 192 - 60 T)

Specs. for Alloy-Steel Bolting Materials for High-Temperature Service (A 193 - 60 T)

Specs. for Seamless Cold-Drawn Intermediate Alloy-Steel Heat Exchanger and Condenser Tubes (A 199 - 60 T)

Specs. for Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (A 200 - 60 T)

Spec. for Carbon-Silicon Steel Plates for Intermediate Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels (A 201 - 57 T) with the deletion of the revision of Section 9.

Specs. for Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 209 - 60 T)

Specs. for Medium-Carbon Seamless Steel Boiler and Superheater Tubes (A 210 - 60 T)

Spec. for High Tensile Strength Carbon-Silicon Steel Plates for Boilers and Other Pressure Vessels (A 212 - 57 T) with the deletion of the revision of Section 9.

Specs. for Seamless Alloy Steel Boiler, Superheater, and Heat Exchanger Tubes (A 213 - 60 T)

Specs. for Electric-Resistance-Welded Steel Heat-Exchanger and Condenser Tubes (A 214 - 60 T)

Specs. for Electric-Resistance-Welded Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 - 60 T)

Specs. for Flat-Rolled Carbon Steel Sheets of Structural Quality (A 245 - 58 T)

Specs. for Welded Austenitic Stainless Steel Boiler, Superheater, Heat Exchanger, and Condenser Tubes (A 249 - 60 T)

Specs. for Electric-Resistance-Welded Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 250 - 60 T)

Spec. for Alloy Steel Bars to End Quench Hardenability Requirements (A 304 - 57 T)

Spec. for Low-Carbon Steel Externally and Internally Threaded Standard Fasteners (A 307 - 61 T)

Spec. for Stress Relief Annealed Cold-Drawn Carbon Steel Bars (A 311 - 60 T)

Specs. for Seamless and Welded Austenitic Stainless Steel Pipe (A 312 - 60 T) (Jointly with Committee A-10)

Spec. for Quenched and Tempered Carbon Steel Bars (A 321 - 60 T)

Spec. for Hot-Rolled Alloy Steel Bars (A 322 - 57 T)

Spec. for Cold-Finished Alloy Steel Bars (A 331 - 57 T)

Specs. for Seamless and Welded Steel Pipe for Low-Temperature Service (A 333 - 60 T)
Specs. for Seamless and Welded Steel Tubes for Low-Temperature Service (A 334 - 60 T)
Specs. for Seamless Ferritic Alloy Steel Pipe for High-Temperature Service (A 335 - 60 T)
Spec. for Alloy Steel Seamless Drum Forgings (A 336 - 57 T)
Specs. for Forged or Rolled Carbon and Alloy Steel Flanges, Forged Fittings, and Valves and Parts for Low-Temperature Service (A 350 - 59 T)
Specs. for Ferritic and Austenitic Steel Castings for High-Temperature Service (A 351 - 60 T)
Specs. for Ferritic Alloy Steel Forged and Bored Pipe for High-Temperature Service (A 369 - 60 T)
Methods and Definitions for Mechanical Testing of Steel Products (A 370 - 54 T)
Specs. for High-Strength Low Alloy Hot-Rolled Steel Sheets and Strip (A 375 - 58 T)
Specs. for Seamless Austenitic Steel Pipe for High-Temperature Central-Station Service (A 376 - 60 T)
Spec. for Chromium-Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 387 - 57 T)
Specs. for Galvanized Steel Transmission Tower Bolts and Nuts (A 394 - 61 T)
Specs. for Factory-Made Wrought Austenitic Steel Welding Fittings (A 403 - 60 T), with the deletion of the new Grade WP 348H from Table I.
Specs. for Seamless Ferritic Alloy-Steel Pipe Specially Heat-Treated for High-Temperature Service (A 405 - 56 T)
Specs. for Factory-Made Wrought Carbon Steel and Alloy Steel Welding Fittings of Seamless or Welded Construction for Low-Temperature Service (A 420 - 59 T)
Specs. for Butt Welds in Still Tubes for Refinery Service (A 422 - 58 T)
Specs. for Seamless and Electric Welded Low-Alloy Steel Tubes for Economizers (A 423 - 60 T)
Specs. for Centrifugally Cast Ferritic Alloy Steel Pipe for High-Temperature Service (A 426 - 59 T)
Specs. for Austenitic Steel Forged and Bored Pipe for High-Temperature Service (A 430 - 60 T)

Adopted as Standard:

Method of Magnetic Particle Testing and Inspection of Heavy Steel Forgings (A 275 - 58 T)
Spec. for Carbon Steel Axles, Non-Heat-Treated, for Railway Use (A 21 - 57 T)
Spec. for Carbon Steel Forgings for Railway Use (A 236 - 57 T)
Spec. for Alloy Steel Forgings for Railway Use (A 238 - 58 T)
Spec. for Untreated Carbon Steel Axles for Export and for General Industrial Use (A 383 - 57 T)
Spec. for Wrought Alloy Steel Rolls for Cold and Hot Reduction (A 427 - 58 T)
Spec. for Carbon-Chromium Ball and Roller Bearing Steel (A 295 - 46 T)

Adopted as Standard, Revisions in:

Specs. for Steel Screw Spikes (A 66 - 54)
Spec. for Structural Steel for Ships (A 131 - 59)
Specs. for Electric-Fusion (Arc)-Welded Steel Plate Pipe (Sizes 16 in. and Over) (A 134 - 54)
Specs. for Electric-Fusion (Arc)-Welded Steel Pipe (Sizes 4 in. and Over) (A 139 - 58)
Spec. for Nickel-Steel Plates for Boilers and Other Pressure Vessels (A 203 - 60) with the deletion of the revision of Section 9.
Spec. for Molybdenum-Steel Plates for Boilers and Other Pressure Vessels (A 204 - 57)
Specs. for Welded and Seamless Steel Pipe Piles (A 252 - 59)
Specs. for Copper Brazed Steel Tubing (A 254 - 58)
Specs. for Seamless and Welded Ferritic Stainless Steel Tubing for General Service (A 268 - 60)
Specs. for Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 - 60)
Spec. for Seamless and Welded Austenitic Stainless Steel Sanitary Tubing (A 270 - 60)
Spec. for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 - 60)
Spec. for Metal-Arc Welded Steel Pipe for High-Pressure Transmission Service (A 381 - 58)

Standard Withdrawn:

Specs. for Welded and Seamless Open-Hearth Iron Pipe (A 253 - 60)

Committee A-1 withdrew from the preprinted report recommendations for the revision of Tentative Specifications A 273, A 274, and A 410, and revisions for immediate adoption of Standard Specification A 353.

Committee A-5 on Corrosion of Iron and Steel:

The report was presented by H. F. Hormann, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Copper Covered Steel Wire Strand (A 460 - 61 T)
Spec. for Zinc-Coated Flat Steel Armoring Tape (A 459 - 61 T)

Adopted as Standard, Revisions in:

Specs. for Zinc Coating (Hot Dip) on Iron and Steel Hardware (A 153 – 60)

Specs. for Zinc Coating (Hot Dip) on Assembled Steel Products (A 386 – 59)

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys:

Report presented by L. L. Wyman, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Hot and Cold-Worked Alloy Steel Plate, Sheet, and Strip for High Strength at Elevated Temperatures (A 457 – 61 T)

Spec. for Hot and Cold-Worked Alloy Steel Bars for High Strength at Elevated Temperatures (A 458 – 61 T)

Spec. for Centrifugally Cast Austenitic Steel Pipe for High-Temperature Service (A 451 – 61 T) (Jointly with Committee A-1)

Spec. for Centrifugally Cast Austenitic Cold-Wrought Pipe for High-Temperature Service (A 452 – 61 T) (Jointly with Committee A-1)

Spec. for High-Strength, High-Temperature Bolting Materials with Expansion Coefficients Comparable to Austenitic Steels (A 453 – 61 T) (Jointly with Committee A-1)

Accepted as Tentative, Revisions in:

Spec. for Seamless and Welded Austenitic Stainless Steel Pipe (A 312 – 60 T) (Jointly with Committee A-1)

Spec. for Corrosion-Resisting Chromium Steel Clad Plate, Sheet, and Strip (A 263 – 59 T)

Spec. for Corrosion-Resisting Chromium-Nickel Steel Clad Plate, Sheet, and Strip (A 264 – 59 T)

Spec. for Corrosion-Resistant Iron-Chromium and Iron-Chromium-Nickel Alloy Castings for General Application (A 296 – 60 T)[18a]

Adopted as Standards, Revisions in:

Spec. for Seamless and Welded Ferritic Stainless Steel Tubing for General Service (A 268 – 60) (Jointly with Committee A-1)

Spec. for Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 – 60) (Jointly with Committee A-1)

Spec. for Seamless and Welded Austenitic Stainless Steel Sanitary Tubing (A 270 – 60) (Jointly with Committee A-1)

Spec. for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 – 60) (Jointly with Committee A-1)

Committee B-2 on Non-Ferrous Metals and Alloys:

Report presented by Alfred Bornemann, chairman, and the following actions taken:

Adopted as Standard:

Spec. for White Metal Bearing Alloys (Known Commercially as "Babbitt Metal") (B 23 – 59 T)

Spec. for Nickel-Copper Alloy Plate, Sheet, and Strip (B 127 – 58 T)

Spec. for Nickel Rod and Bar (B 160 – 58 T)

Spec. for Nickel Seamless Pipe and Tube (B 161 – 58 T)

Spec. for Nickel Plate, Sheet, and Strip (B 162 – 58 T)

Spec. for Nickel-Copper Alloy Rod and Bar (B 164 – 58 T)

Spec. for Nickel-Copper Alloy Seamless Pipe and Tube (B 165 – 58 T)

Committee B-5 on Copper and Copper Alloys, Cast and Wrought:

Report presented by W. H. Jennings, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Copper-Nickel Alloy Castings (B 369 – 61 T)

Adopted as Standard:

Spec. for Copper Sheet and Strip for Building Construction (B 370 – 61)

Spec. for Copper-Zinc-Silicon Alloy Rod (B 371 – 61)

Spec. for Seamless Copper and Copper-Alloy Rectangular Waveguide Tube (B 372 – 61)

Spec. for Bronze Castings for Bridges and Turntables (B 22 – 60 T)

Spec. for General Requirements for Wrought Copper-Alloy Wire (B 250 – 60 T)

Spec. for Flat Copper Products with Finished (Rolled or Drawn) Edges (Flat Wire and Strip) (B 272 – 60 T)

Adopted as Standard, Revisions in:

Spec. for Phosphor Bronze Plate, Sheet, Strip, and Rolled Bar (B 103 – 60)

Spec. for Copper Plates for Locomotive Fireboxes (B 11 – 58)

Spec. for Copper Rods for Locomotive Staybolts (B 12 – 58)

Spec. for Cartridge Brass Sheet, Strip, Plate, Bar, and Disks (B 19 – 58)

Spec. for Brass Plate, Sheet, Strip, and Rolled Bar (B 36 – 60)

Spec. for Seamless Copper Tube (B 75 – 60)

Spec. for Seamless Copper Water Tube (B 88 – 58)

Spec. for Copper-Silicon Alloy Plate and Sheet for Pressure Vessels (B 96 – 58)

[18a] Publication of the revision was withheld pending the approval by confirming letter ballot vote in the committee.

Spec. for Copper-Silicon Alloy Wire for General Purposes (B 99 – 54)
Spec. for Rolled Copper-Alloy Bearing and Expansion Plates and Sheets for Bridge and Other Structural Uses (B 100 – 58)
Spec. for Commercial Bronze Strip (B 130 – 58)
Spec. for Seamless Brass Tube (B 135 – 60)
Spec. for Tin Bronze and Leaded Tin Bronze Sand Castings (B 143 – 52)
Spec. for Leaded Red Brass and Leaded Semi-Red Brass Sand Castings (B 145 – 52)
Spec. for Seamless Copper Bus Pipe and Tube (B 188 – 60)
Spec. for General Requirements for Wrought Copper and Copper-Alloy Plate, Sheet, Strip, and Rolled Bar (B 248 – 59)
Spec. for General Requirements for Wrought Copper and Copper-Alloy Rod, Bar, and Shapes (B 249 – 60)
Spec. for General Requirements for Wrought Seamless Copper and Copper-Alloy Pipe and Tube (B 251 – 60)
Spec. for Copper-Zinc-Manganese Alloy (Manganese Brass) Sheet and Strip (B 291 – 60)
Spec. for Threadless Copper Pipe (B 302 – 60)
Spec. for Copper Drainage Tube (DWV) (B 306 – 59)
Spec. for Copper-Silicon Alloy Seamless Pipe and Tube (B 315 – 59)

Standard Withdrawn:

Spec. for Seamless Copper Boiler Tubes (B 13 – 58)

Committee D-1 on Paint, Varnish, Lacquer and Related Products:

Report presented by W. T. Pearce, chairman, and the following actions taken:

Accepted as Tentative:

Specs. for Distilled Coconut Fatty Acids (D 1841 – 61 T)
Specs. for Distilled Corn Fatty Acids (D 1842 – 61 T)
Specs. for Fractionated and Distilled Cottonseed Fatty Acids (D 1843 – 61 T)
Spec. for Asbestos-Cement Shingle Blanks to Be Used as Panels in Weathering Tests of Latex and Emulsion Paints (D 1911 – 61 T)
Methods for Chemical Analysis of Strontium Chromate Pigment (D 1845 – 61 T)
Test for Water-Soluble Matter in Iron Oxide Pigments by Specific Resistance Method (D 1846 – 61 T)
Methods for Chemical Analysis of Basic Lead Silico-Chromate (D 1844 – 61 T)
Test for Total Chlorine Content of Epoxy Resins (D 1847 – 61 T)
Method of Reporting Paint Film Failures Characteristic of Exterior Latex Paints (D 1848 – 61 T)
Test for Package Stability of Latex Paint (D 1849 – 61 T)
Test for Hydroxyl Value of Fatty Oils and Acids (D 1957 – 61 T) (This test was listed in error in the preprinted report as a new standard.)

Accepted as Tentative, Revisions in:

Test for Acidity in Lacquer Solvents and Diluents (D 1613 – 58 T)
Def. of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 – 59)

Adopted as Standard:

Spec. for Raw Tung Oil (D 12 – 59 T)
Spec. for Boiler Linseed Oil (D 260 – 58 T)
Spec. for Basic Lead Silico-Chromate (D 1648 – 59 T)
Test for Viscosity Reduction Power of Hydrocarbon Solvents (D 1311 – 54 T)
Test for No-Smear Time of Traffic Paint (D 1359 – 55 T)
Method of Sampling Liquid Oils and Fatty Acids Commonly Used in Paints, Varnishes, and Related Materials (D 1466 – 57 T)
Test for Effect of Staining Agents on Organic Finishes Used in the Transportation Industry (D 1540 – 58 T)
Test for Phthalic Acid Isomers and Benzoic Acid in Alkyd Resins and Esters (D 1651 – 59 T)
Test for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments (D 1654 – 59 T)
Test for Kauri-Butanol Value of Solvents (D 1133 – 54 T), as revised
Methods of Testing Fatty Acids Used in Protective Coatings (D 1467 – 59 T), as revised
Test for Specific Gravity of Drying Oils, Varnishes, Resins, and Related Materials at 25/25 C (D 1963 – 61)
Test for Saponification Value of Drying Oils and Fatty Acids (D 1962 – 61)
Test for Unsaponifiable Matter in Drying Oils and Fatty Acids (D 1965 – 61)
Test for Iodine Value of Drying Oils and Fatty Acids (D 1959 – 61)
Test for Loss on Heating of Drying Oils (D 1960 – 61)
Test for Foots in Raw Linseed Oil (D 1954 – 61)
Test for Quantitative Determination of Break in Drying Oils (D 1952 – 61)
Test for Acetone Tolerance of Heat-Bodied Drying Oil (D 1950 – 61)
Test for Gel Time of Drying Oils (D 1955 – 61)
Test for Tung Oil Quality (D 1964 – 61)
Test for Drying Properties of Drying Oils (D 1953 – 61)
Test for Ash in Drying Oils and Fatty Acids (D 1951 – 61)
Test for Matter Insoluble in Chloroform in Oiticica Oil (D 1958 – 61)

Test for Heat Bodying Rate of Drying Oils (D 1956 - 61)
Test for Maleic Diene Value of Drying Oils (D 1961 - 61)
Test for Titer of Fatty Acids (D 1982 - 61)
Test for Acid Value of Fatty Acids (D 1980 - 61)
Test for Color After Heating of Fatty Acids (D 1981 - 61)

Adopted as Standard, Revisions in:

Test for Flash Point by Tag Closed Tester (D 56 - 56)
Methods of Sampling and Testing Lacquer Solvents and Diluents (D 268 - 59)
Specs. for Leaded Oxide (D 80 - 41)
Specs. for Petroleum Spirits (Mineral Spirits) (D 235 - 59)
Tests for Nonvolatile Content of Resin Solutions (D 1259 - 58)
Specs. for Industrial 90 Benzene for Use in Paint, Varnish, Lacquer, and Related Products (D 361 - 36)
Specs. for Industrial Grade Toluene for Use in Paint, Varnish, Lacquer, and Related Products (D 362 - 36)
Specs. for Industrial Grade Xylene or Solvent Naphtha for Use in Paint, Varnish, Lacquer, and Related Products (D 364 - 36)
Methods of Testing Drying Oils (D 555 - 58)
Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products (D 609 - 52)
Test for Acid Value of Varnishes (D 1639 - 59)

Standards Withdrawn:

Test for Relative Dry Hiding Power of White Pigments in a Linseed Oil Vehicle (D 406 - 39). This method has been superseded by Method D 1738.
Test for Nitrocellulose Diluting Power of Hydrocarbon Solvents (D 1134 - 53). This method is now included in Method D 1720.

Tentative Withdrawn:

Spec. for Heavy Petroleum Spirits (Heavy Mineral Spirits) (D 965 - 59 T)

The committee withdrew from the preprinted report the recommendation for adoption as standard without revision of the Tentative Specifications for Raw Linseed Oil (D 234 - 58 T).

There failed of approval at the session the recommendation of Committee D-1 for the publication as tentative of the proposed Method of Test for Perspiration Resistance of Organic Coatings. The method is to be given further consideration in the committee in the light of the objections raised at the session.

Committee D-5 on Coal and Coke:

Report presented by R. L. Coryell, chairman, and the following actions taken:

Accepted as Tentative:

Test for Fusibility of Coal Ash (D 1857 - 61 T)

Accepted as Tentative, Revisions in:

Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 - 58)

Adopted as Standard:

Test for Equilibrium Moisture of Coal at 96 to 97 Per Cent Relative Humidity and 30 C (D 1412 - 56 T)

Committee D-17 on Naval Stores:

Report presented by S. R. Snider, chairman, and the following actions taken subject to a favorable vote in the committee on the report:

Adopted as Standard, Revisions in:

Methods of Testing Tall Oil (D 803 - 58)

The revision of Standard Methods of Sampling and Grading Rosin (D 509 - 55) was inadvertently listed in the report as a substance change for immediate adoption instead of as an editorial change.

Committee E-16 on Sampling and Analysis of Metal Bearing Ores and Related Materials:

Report presented by J. L. Hague, chairman, and accepted as a report of progress.

TWENTY-NINTH SESSION—COMMITTEE REPORT SESSION

WEDNESDAY, JUNE 28, 4:30 P.M.

SESSION CHAIRMAN: R. F. LEGGET

Committee C-7 on Lime:

Report presented in the absence of the chairman by Kenneth Gutschick, secretary elect, and the following action taken:

Adopted as Standard, Revisions in:

Spec. for Hydraulic Hydrated Lime for Structural Purposes (C 141 – 55)

Committee C-9 on Concrete and Concrete Aggregates:

Report presented by Bryant Mather, chairman, and the following actions taken:

Accepted as Tentative:

Test for Static Young's Modulus of Elasticity and Poisson's Ratio in Compression of Cylindrical Concrete Specimens (C 469 – 61 T)

Spec. for Single-Use Molds for Forming 6 by 12-in. Concrete Compression Test Cylinders (C 470 – 61 T), with the addition of the following new Paragraph (*d*) to Section 4:

(*d*) Calculate the elongation as the difference between the final length and initial length. Calculate the absorption as the difference between the final weight and the initial weight of the sample mold.

Accepted as Tentative, Revisions in:

Test for Rate of Hardening of Mortars Sieved from Concrete Mixtures by Proctor Penetration Resistance Needles (C 403 – 57 T)

Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88 – 59 T)

Test for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar Bar Method) (C 227 – 60 T)

Test for Potential Reactivity of Aggregates (Chemical Method) (C 289 – 57 T)

Test for Resistance of Concrete Specimens to Rapid Freezing and Thawing in Water (C 290 – 57 T)

Test for Resistance of Concrete Specimens to Rapid Freezing in Air and Thawing in Water (C 291 – 57 T)

Test for Resistance of Concrete Specimens to Slow Freezing and Thawing in Water or Brine (C 292 – 57 T)

Test for Resistance of Concrete Specimens to Slow Freezing in Air and Thawing in Water (C 310 – 57 T)

Test for Potential Volume Change of Cement-Aggregate Combinations (C 342 – 55 T)

Methods of Sampling and Testing Fly Ash as an Admixture in Portland Cement Concrete (C 311 – 59 T)

Methods of Securing, Preparing, and Testing Specimens from Hardened Concrete for Compressive and Flexural Strengths (C 42 – 60)

Specs. for Ready-Mixed Concrete (C 94 – 58)

Specs. for Concrete Aggregates (C 33 – 59) (Standard reverted to tentative)

Test for Amount of Material Finer Than No. 200 Sieve in Aggregate (C 117 – 49) (Standard reverted to tentative) (Jointly with Committee D-4)

Test for Sieve Analysis of Fine and Coarse Aggregates (C 136 – 46) (Standard reverted to tentative) (Jointly with Committee D-4)

Adopted as Standard:

Spec. for Lightweight Aggregates for Insulating Concrete (C 332 – 56 T)

Spec. for Cotton Mats for Curing Concrete (C 440 – 59 T)

Adopted as Standard, Revisions in:

Test for Compressive Strength of Molded Concrete Cylinders (C 39 – 59)

Methods of Securing, Preparing, and Testing Specimens from Hardened Concrete for Compressive and Flexural Strengths (C 42 – 60)

Specs. for Ready-Mixed Concrete (C 94 – 58)

Committee C-12 on Mortar for Unit Masonry:

Report presented by H. C. Plummer, chairman, and the following actions taken:

Accepted as Tentative:

Specs. for Mortar and Grout for Reinforced Masonry (C 476 – 61 T)

Accepted as Tentative, Revisions in:

Specs. for Mortar for Unit Masonry (C 270 – 59 T)

Adopted as Standard:

Specs. for Aggregates for Masonry Grout (C 404 – 57 T)

Committee C-13 on Concrete Pipe:

Report presented by R. R. Litehiser, chairman, and the following actions taken:

Accepted as Tentative:

Specs. for Precast Reinforced Concrete Manhold Risers and Tops (C 478 – 61 T)
Specs. for Cast-in-Plate Nonreinforced Concrete Irrigation Closed Conduit (C 477 – 61 T)

Accepted as Tentative, Revisions in:

Spec. for Reinforced Concrete Culvert, Storm Drain, and Sewer Pipe (C 76 – 60 T)

Adopted as Standard:

Specs. for Reinforced Concrete Low-Head Pressure Pipe (C 361 – 59 T)

Committee D-8 on Bituminous Materials for Roofing, Waterproofing, and Related Building or Industrial Uses:

Report presented by H. R. Snoke, chairman, and the following actions taken:

Accepted for Publication as Information Only:

Proposed Method for Defining and Measuring the Temperature Susceptibility of Asphalts

Accepted as Tentative:

Specs. for Homogeneous Bituminized Fiber Drain and Sewer Pipe (D 1861 – 61 T)
Specs. for Laminated-Wall Bituminized Fiber Drain and Sewer Pipe (D 1862 – 61 T)
Specs. for Mineral Aggregate for Use on Built-Up Roofs (D 1863 – 61 T)
Test for Moisture in Mineral Aggregate for Use on Built-Up Roofs (D 1864 – 61 T)
Test for Hardness of Mineral Aggregate for Use on Built-Up Roofs (D 1865 – 61 T)
Test for Translucency of Mineral Aggregate for Use on Built-Up Roofs (D 1866 – 61 T)

Accepted as Tentative, Revisions in:

Spec. for Woven Glass Fabrics Saturated with Bituminous Substances for Use in Waterproofing (D 1668 – 59 T)
Spec. for Primer for Use with Asphalt in Dampproofing and Waterproofing (D 41 – 41)
Spec. for Asphalt Shingles Surfaced with Mineral Granules (D 225 – 60)

Adopted as Standard, Revisions in:

Test for Steam Distillation of Bituminous Protective Coatings (D 255 – 28)

Standard Withdrawn:

Spec. for Sieve Analysis of Granular Mineral Surfacing for Asphalt Roofing and Shingles (D 1001 – 51)

Committee D-18 on Soils for Engineering Purposes:

Report presented in the absence of the chairman by A. A. Wagner, secretary, and the following actions taken:

Accepted as Tentative:

Test for Bearing Ratio of Laboratory-Compacted Soils (D 1883 – 61 T)

Accepted as Tentative, Revisions in:

Method for Grain Size Analysis of Soils (D 422-60 T)
Test for Liquid Limit of Soils (D 423 – 59 T)
Test for Load-Settlement Relationship for Individual Piles Under Vertical Axial Load (D 1143 – 57 T)

Adopted as Standard:

Methods of Testing Soil-Bituminous Fixtures (D 915 – 47 T)

Adopted as Standard, Revisions in:

Test for Shrinkage Factors of Soils (D 427 – 39)

Committee E-6 on Methods of Testing Building Constructions:

Report presented by R. W. Bletzacker, membership secretary, and the following actions taken:

Adopted as Standard, Revisions in:

Methods of Conducting Strength Tests of Panels for Building Construction (E 72 – 55)

The committee withdrew from the preprinted report the recommendation for reapproval of the Recommended Practice for Laboratory Measurement of Airborne-Sound Transmission Loss of Building Floors and Walls (E 90 – 55) since a revision of the recommended practice will be presented to the Society through the Administrative Committee on Standards.

6:30 P.M. COCKTAIL PARTY

AND

7:30 P.M. ANNUAL DINNER

WEDNESDAY, JUNE 28

The Philadelphia District, through its Council, acted as the official host at the Cocktail Party and Dinner which was followed by entertainment. Approximately 294 persons attended this function.

THIRTIETH SESSION—SYMPOSIUM ON ELEVATED-TEMPERATURE COMPRESSION TESTING OF SHEET MATERIALS[19]

THURSDAY, JUNE 29, 9:00 A.M.

SESSION CHAIRMAN: H. C. TURNER

Introduction by H. C. Turner.

An Evaluation of Compression Testing Techniques of Sheet Materials at Elevated Temperatures—George Gerard, presented by Ralph Papirno.

Compression Testing of Sheet Materials at Elevated Temperatures—Ralph Papirno and George Gerard, presented by Mr. Papirno.

Rapid Rate Compression Testing of Sheet Materials at High Temperatures—E. C. Bernett and W. W. Gerberich, presented by Mr. Bernett.

Evaluation of Test Variables in the Determination of Elevated Temperature Compression Yield Strength of Magnesium Sheet—R. W. Fenn, presented by the author.

Compression and Buckling Characteristics of Annealed and Aged Inconel 718 Nickel-Chromium Alloy at Temperatures up to 1400 F—Julian Dubuc and Georges Welter, presented by Mr. Dubuc.

An Evaluation of a System for the Compression Testing of Sheet Materials at Elevated Temperatures—W. W. Breindel, R. L. Carlson, and F. C. Holden, presented by Mr. Breindel.

Summary by J. R. Kattus.

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[19] Issued as *ASTM STP No. 303.*

THIRTY-FIRST SESSION—SESSION ON GENERAL TESTING

THURSDAY, JUNE 29, 9:30 A.M.

SESSION CHAIRMAN: VICE-PRESIDENT-ELECT A. C. WEBBER

Stress-Relaxation—Some New Test Methods for the Determination of This Mechanical Property Either in Tension or in Compression—G. R. Gohn and A. Fox, presented by Mr. Gohn.[20]

The Effect of Specimen Geometry on Determination of Elongation in Sheet Tension Specimens—E. B. Kula and N. H. Fahey, presented by Mr. Kula.[21]

The Effect of Section Size on the Notch Strength and Fracture Development in Selected Structural Metals—E. P. Klier and Volker Weiss, presented by Mr. Klier.

Potential Heat: A Method for Measuring the Heat Release of Materials in Building Fires—J. J. Loftus, D. Gross, and A. F. Robertson, presented by Mr. Loftus.

A Method for Determining the Plastic Flow Properties of Sheet and Round Tension Specimens—John Nunes and F. R. Larson, presented by Mr. Nunes.

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[20] Published in ***Materials Research & Standards,*** Vol. 1, No. 12, p. 957, Dec., 1961.

[21] Published in ***Materials Research & Standards,*** Vol. 1, No. 8, p. 631, Aug., 1961.

CHEMICAL AND MECHANICAL PROPERTIES OF ASBESTOS-CEMENT PIPE[22]

THURSDAY, JUNE 29, 9:30 A.M.

The following papers were presented at an informal session sponsored by Committee C-17 on Asbestos-Cement Products.

The Behavior of Normal and Autoclave Cured Asbestos-Cement Pipe Under Various Sulfate Conditions During Long-Term Field and Laboratory Test—L. R. Blair and Philip W. Manson.

Preliminary Research Results of Chemical Tests and Mechanical Properties of Asbestos-Cement Pipe—Paul Brennan and N. L. Menerow.

The Mechanism of Sulfate Attack on Portland Cement Paste—William Lerch.

THIRTY-SECOND SESSION—COMMITTEE REPORT SESSION

THURSDAY, JUNE 29, 11:30 A.M.

SESSION CHAIRMAN: PRESIDENT A. A. BATES

Committee C-2 on Magnesium Oxychloride and Magnesium Oxysulfate Cements:

Report presented in the absence of the chairman by R. E. Hess, ASTM Staff, and the following action taken:

Adopted as Standard:

Spec. for Oxychloride Magnesia (C 275 - 56 T)

Committee C-3 on Non-Metallic Protective Materials:

Report presented in the absence of the chairman by R. E. Hess, ASTM Staff, and the following actions taken:

Accepted as Tentative:

Spec. for Chemically Setting Silicate-Type Chemical-Resistant Mortars (C 466 - 61 T)

Accepted as Tentative, Revisions in:

Tests for Working and Initial Setting Times of Chemically Setting, Silicate-Type Chemical-Resistant Mortars (C 414 - 58 T)

Adopted as Standard, Revisions in:

Test for Tensile Strength of Resin-Type Chemical-Resistant Mortars (C 307 - 55)

Tests for Working and Setting Times of Resin-Type Chemical-Resistant Mortars (C 308 - 55)

Committee D-7 on Wood:

Report presented in the absence of the chairman by R. E. Hess, ASTM Staff, and the following actions taken:

Accepted as Tentative:

Spec. for Petroleum for Blending with Creosote (D 1859 - 61 T)

Spec. for Creosote-Petroleum Solution (D 1858 - 61 T)

Test for Moisture and Creosote-Type Preservative in Wood (D 1860 - 61 T)

Accepted as Tentative, Revisions in:

Spec. for Pressure Treatment of Timber Products (D 1760 - 60 T)

Spec. for Creosote - Coal Tar Solution (D 391 - 53) (Standard reverted to tentative)

Adopted as Standard:

Spec. for Acid Copper Chromate (D 1624 - 59 T)

Spec. for Chromated Copper Arsenate (D 1625 - 59 T)

Spec. for Chromated Zinc Arsenate (D 1626 - 59 T)

Methods for Chemical Analysis of Acid Copper Chromate (D 1627 - 59 T)

Methods for Chemical Analysis of Chromated Copper Arsenate (D 1628 - 59 T)

Methods for Chemical Analysis of Chromated Zinc Arsenate (D 1629 - 59 T)

Method of Testing Wood Preservatives by Laboratory Soil-Block Cultures (D 1413 - 56 T)

Standards Withdrawn:

Methods of Chemical Analysis of Zinc Chloride (D 199 - 50)

Spec. for Zinc Chloride (D 432 - 50)

Committee D-10 on Packaging:

Report presented in the absence of the chairman by R. E. Hess, ASTM Staff, and the following action taken subject to a favorable vote in the committee on the report:

Adopted as Standard:

Drop Test for Shipping Containers (D 775 - 57 T)

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[22] To be published in *Materials Research & Standards.*

Committee D-12 on Soaps and Other Detergents:

Report presented in the absence of the chairman by R. E. Hess, ASTM Staff, and accepted as a report of progress.

Committee D-13 on Textile Materials:

Report presented by H. F. Schiefer, chairman, and the following actions taken:

Accepted for Publication as Information Only:

Proposed Test for Length and Length Distribution of Cotton Fibers by the Short Method Array

Accepted as Tentative:

Test for Dimensional Change in Woven and Knitted Textiles (Excluding Wool) (D 1905 – 61 T)
Test for Estimating Maturity and Weight per Unit Length of Cotton Fibers by the Causticaire Method (D 2480 – 61 T)
Test for Estimation of Effective Gage Lengths in Single Fibers Testing (D 1906 – 61 T)
Test for Moisture Content and Moisture Regain of Lint Cotton and Seed Cotton (Oven Method) (D 2495 – 61 T)
Test for pH of Aqueous Extracts of Wool and Similar Animal Fibers (D 2165 – 61 T)
Test for Rapid Estimation of Staple Length of Wool Top (D 2142 – 61 T)
Test for Yarn Number (Skein Method) (D 1907 – 61 T)
Test for Yarn Severance in Woven Fabrics (D 1908 – 61 T)
Test for Colorfastness of Zipper Stringers to Dry Cleaning (D 2052 – 61 T)
Test for Colorfastness of Zipper Tapes to Crocking (D 2054 – 61 T)
Test for Colorfastness of Zipper Tapes to Perspiration (D 2055 – 61 T)
Test for Colorfastness of Zippers to Laundering (D 2051 – 61 T)
Test for Colorfastness of Zippers to Light (D 2053 – 61 T)
Test for Durability of Finish of Zippers to Dry Cleaning (D 2058 – 61 T)
Test for Durability of Finish of Zippers to Laundering (D 2057 – 61 T)
Methods for Measuring Zippers (D 2060 – 61 T)
Test for Operability of Zippers (D 2062 – 61 T)
Test for Resistance of Finish of Zippers to Dry Abrasion (D 2056 – 61 T)
Test for Resistance of Zippers to Salt Fog (D 2059 – 61 T)
Methods for Strength Tests of Zippers (D 2061 – 61 T)
Def. of Terms Relating to Zippers (D 2050 – 61 T)
Listing of Commercial Moisture Regains for Textile Fibers (D 1909 – 61 T)
Rec. Practice for Establishment of Standard Moisture Content for Wool and Its Products (D 2118 – 61 T)

Accepted as Tentative, Revisions in:

Methods of Testing and Tolerances for Cotton Yarns (D 180 – 57 T)
Methods for Identification of Fibers in Textiles (D 276 – 60 T)
Test for Abrasion Resistance of Textile Fabrics (D 1175 – 55 T)
Test for Average Fiber Diameter of Wool Tops, Card Sliver, and Scoured Wool by Micronaire Method (D 1282 – 57 T)
Methods of Testing Tufted Pile Floor Coverings (D 1486 – 57 T)
Methods of Testing and Tolerances for Yarns Containing Wool (D 1285 – 58 T)
Methods of Testing Spun and Filament Yarns Made Wholly or in Part of Man-Made Organic Base Fibers (D 1380 – 57 T)
Test for Elastic Properties of Uncrimped Fibers (D 1774 – 60 T)
Method of Conditioning Textiles and Textile Products for Testing (D 1776 – 60 T)
Def. of Terms Relating to Textile Materials (D 123 – 60)

Adopted as Standard:

Test for Diameter of Wool and Mohair Fibers by Microprojection (D 2130 – 61)
Rec. Practice for Lighting Cotton Classing Rooms for Color Grading (D 1684 – 59 T)
Test for Heat Aging of Asbestos Textiles (D 1573 – 58 T)
Test for Fiber Length of Wool (D 1575 – 58 T)
Test for Flammability of Clothing Textiles (D 1230 – 52 T), as revised

Adopted as Standard, Revisions in:

General Methods of Textile Woven Fabrics (D 39 – 59): covering (1) adoption as standard of tentative revisions of Sections 3 to 14 and 17 and 18; and (2) immediate adoption of general revisions.
Spec. and Test for Fineness of Wool (D 419 – 58)
Methods of Testing Felt (D 461 – 59)
Spec. and Test for Fineness of Wool Tops (D 472 – 56)
Methods of Testing and Tolerances for Glass Yarns (D 578 – 58)
Spec. and Test for Fineness of Mohair Tops (D 1381 – 59)

Committee D-13 withdrew from the preprinted report recommendations for the revision of Tentative Method of Test D 39 – 59 T; revision of Tentative Methods of Test D 1175 – 55 T covering the addition of new Sections 35 to 41 on the accelerator abrasion tester; tentative revision of Standard Definitions D 123 - 60 covering the deletion of the definition for "Condition, Standard" in Part A; and the revision for immediate adoption of Standard Specifications D 1114 - 51.

Committee E-1 on Methods of Testing:

Report presented in the absence of the chairman by W. J. McCoy, vice-chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for ASTM Thermometers (E 1 - 60) (This recommendation, covering the addition of two new Saybolt Furol Viscosity Thermometers, 108 F and 109 F was inadvertently listed in the preprinted report as a new tentative.)
Methods of Verification of Testing Machines (E 4 - 60 T)
Spec. for ASTM Hydrometers (E 100 - 59 T)

Adopted as Standard:

Def. of Terms Relating to Methods of Mechanical Testing (E 6 - 60 T)
Method for Determination of Young's Modulus at Room Temperature (E 111 - 59 T), as revised
Test for Indentation Hardness of Metallic Materials by Portable Hardness Testers (E 110 - 55 T), as revised
Method for Determination of Shear Modulus at Room Temperature (E 143 - 59 T), as revised
Methods of Compression Testing of Metallic Materials (E 9 - 59 T)
Test for Brinell Hardness of Metallic Materials (E 10 - 60 T)
Spec. for Sieves for Testing Purposes (Wire Cloth Sieves, Round-Hole and Square-Hole Screens or Sieves) (E 11 - 60 T)
Test for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials (E 18 - 60 T)
Method of Rapid Indentation Hardness Testing of Metallic Materials (E 103 - 60 T)
Specs. for Weighing and Drying Apparatus for Microchemical Analysis (E 124 - 57 T)
Test for Maximum Pore Diameter and Permeability of Rigid Porous Filters for Laboratory Use (E 128 - 57 T)
Method for Determination of Poisson's Ratio at Room Temperature (E 132 - 58 T)
Specs. for Apparatus for Microdetermination of Nitrogen by Kjeldahl Method (E 147 - 59 T)
Specs. for Apparatus for Microdetermination of Nitrogen by Dumas Method (E 148 - 59 T)

Adopted as Standard, Revisions in:

Specifications for ASTM Thermometers (E 1 - 60): covering (1) adoption as standard of the tentative revision comprising twelve kinematic viscosity thermometer specifications; and (2) immediate adoption of revisions of Thermometers 9 C and 10 C in Table I.

Committee E-12 on Appearance:

Report presented by G. W. Ingle, chairman, and the following action taken:

Accepted as Tentative:

Rec. Practice for Selection of Geometric Conditions for Measurement of Reflectance and Transmittance (E 179 - 61 T)

THIRTY-THIRD SESSION—SYMPOSIUM ON EROSION AND CAVITATION[23]

THURSDAY, JUNE 29, 2:00 P.M.

SESSION CHAIRMAN: R. E. PETERSON

Cavitation in Hydraulic Turbines—W. J. Rheingans, presented by the author.
Liquid Drop Collisions—Olive Engel, presented by the author.
Erosion by Liquid Impact—S. M. DeCorso and R. E. Kothmann, presented by Mr. DeCorso.
Accelerated Cavitation Erosion and Sand Erosion—W. C. Leith and W. S. McIlquham, presented by Mr. McIlquham.
Erosion by Solid Particles in a Fluid Stream—Iain Finnie, presented by the author.
The Mechanism of the Deformation of Solids by the Impact of Liquids at High Speeds—J. H. Brunton, presented by S. M. DeCorso.

PANEL DISCUSSION ON THE EFFECT ON MATERIALS OF NATURALLY OCCURRING SPACE RADIATION[24]

THURSDAY, JUNE 29, 2:00 P.M.

A task group was appointed in 1960 by Committee E-10 on Radioisotopes and Radiation Effects to investigate this subject. There was an extensive report of this investigation as well as discussion.

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[23] Issued as *ASTM STP No. 307.*

[24] To be published as a special technical publication.

THIRTY-FOURTH SESSION—SESSION ON NON-FERROUS METALS

THURSDAY, JUNE 29, 2:30 P.M.

SESSION CHAIRMAN: BRUCE GONSER

Some Effects of Section Size and Testing Condition on the Tensile Properties of Zircaloy 2 at 600 F—S. L. Ames, presented by the author.

Room-Temperature Creep Rate of Molybdenum and Effect of Strain Rate on the Tensile Properties of Molybdenum—R. Q. Barr, M. Semchyshen, and G. A. Timmons, presented by Mr. Semchyshen.

Tensile Strength-Hardness Correlation for Titanium Alloys—C. F. Hickey, Jr., presented by the author.

THIRTY-FIFTH SESSION—COMMITTEE REPORT SESSION

THURSDAY, JUNE 29, 4:30 P.M.

SESSION CHAIRMAN: F. W. REINHART

Committee A-3 on Cast Iron:

Report presented by R. A. Clark, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Gray Iron Castings for Pressure-Containing Parts for Temperatures Up to 650 F (A 278 – 59 T)
Spec. for Austenitic Gray Iron Castings (A 436 – 59 T)
Spec. for Gray Iron Castings for Valves, Flanges, and Pipe Fittings (A 126 – 42) (Standard reverted to tentative)

Adopted as Standard:

Spec. for Cast Iron Culvert Pipe (A 142 – 59 T)
Spec. for Cast Nodular Iron for Pressure Containing Parts for Use at Elevated Temperatures (A 395 – 56 T)

Standards Withdrawn:

Spec. for Lightweight and Thin-Section Gray Iron Castings (A 190 – 47)
Def. of Terms Relating to Cast Iron (A 196 – 47)

Committee A-6 on Magnetic Properties:

Report presented by A. C. Bailer, chairman, and accepted as a report of progress.

Committee B-7 on Light Metals and Alloys, Cast and Wrought:

Report presented by R. A. Harris, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Aluminum Foil for Capacitors (B 373 – 61 T)

Accepted as Tentative, Revisions in:

Spec. for Factory Made Wrought Aluminum and Aluminum-Alloy Welding Fittings (B 361 – 60 T)

Adopted as Standard:

Spec. for Aluminum-Alloy Drawn Seamless Coiled Tubes for Special Purpose Applications (B 307 – 60 T)
Spec. for Aluminum-Alloy Sheet and Plate (B 209 – 60 T)
Spec. for Aluminum-Alloy Drawn Seamless Tubes (B 210 – 60 T)
Spec. for Aluminum-Alloy Bars, Rods, and Wire (B 211 – 60 T)
Spec. for Aluminum-Alloy Extruded Bars, Rods, and Shapes (B 221 – 60 T)
Spec. for Aluminum-Alloy Drawn Seamless Tubes for Condensers and Heat Exchangers (B 234 – 60 T)
Spec. for Aluminum Alloy Extruded Tubes (B 235 – 60 T)
Spec. for Aluminum Bars for Electrical Purposes (Bus Bars) (B 236 – 59 T)
Spec. for Aluminum-Alloy Pipe (B 241 – 60 T)
Spec. for Aluminum-Alloy Die Forgings (B 247 – 60 T)
Spec. for Aluminum-Alloy Standard Structural Shapes, Rolled or Extruded (B 308 – 60 T)
Spec. for Aluminum-Alloy Round Welded Tubes (B 313 – 60 T)
Spec. for Aluminum-Alloy Rivet and Cold Heading Wire and Rods (B 316 – 60 T)
Spec. for Extruded Aluminum-Alloy Bar, Rod, Pipe, and Structural Shapes for Electrical Purposes (B 317 – 60 T)
Spec. for Type A and Type B Aluminum-Alloy Drawn Annealed Seamless Coiled Tubes (B 318 – 60 T)

Spec. for Aluminum-Alloy Pipe for Gas and Oil Transmission and Distribution Piping Systems (B 345 - 60 T)

Adopted as Standard, Revisions in:

Spec. for Magnesium-Base Alloy Sand Castings (B 80 - 59)

Spec. for Magnesium-Base Alloy Sheet and Plate (B 90 - 60)

Spec. for Magnesium Ingot and Stick for Remelting (B 92 - 60)

Spec. for Magnesium-Base Alloy Bars, Rods, and Shapes (B 107 - 60)

Spec. for Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 - 60)

Rec. Practice for Codification of Light Metals and Alloys, Cast and Wrought (B 275 - 59), with the following additional changes:

2. *Basis of Codification.*—(*a*) The designations for alloys and unalloyed metals are based on their chemical composition limits (Note 1).

(*b*) Designations are assigned, revised, and cancelled by Subcommittee VII of ASTM Committee B-7 on Light Metals and Alloys, Cast and Wrought, on written request to its secretary. Complete chemical composition limits shall be submitted with requests for assignment or revision of designations. Arbitrary assignments by other subcommittees or committees will not be recognized.

(*c*) The temper designation, which is used for all metal forms except ingot, follows the alloy designation and is separated therefrom by a dash.

Delete Footnote 3.

Rec. Practice for Temper Designation of Light Metals and Alloys, Cast and Wrought (B 296 - 56)

Committee C-15 on Manufactured Masonry Units:

Report presented in the absence of the chairman by S. F. Etris, ASTM Staff, and the following action taken:

Accepted as Tentative, Revisions in:

Specs. for Ceramic Glazed Structural Clay Facing Tile, Facing Brick, and Solid Masonry Units (C 126 - 60 T)

Committee C-17 on Asbestos-Cement Products:

Report presented by W. V. Friedlaender, chairman, and the following actions taken:

Adopted as Standard:

Spec. and Tests for Corrugated Asbestos-Cement Sheets (C 221 - 58 T)

Adopted as Standard, Revisions in:

Spec. for Flat Asbestos-Cement Sheets (C 220 - 60)

The committee withdrew from the preprinted report the recommendations for publication as tentative of Methods of Testing Asbestos-Cement Pipe, and revisions of Tentative Specification and Tests for Asbestos-Cement Pressure Pipe (C 296 - 59 T) and Tentative Specification and Tests for Asbestos-Cement Non-Pressure Sewer Pipe (C 428 - 59 T).

Committee D-6 on Paper and Paper Products:

Report presented by H. A. Birdsall, chairman, and the following actions taken:

Adopted as Standard, Revisions in:

Test for Reducible Sulfur in Paper (D 984 - 57)

Method of Static Bending Test for Corrugated Paperboard (D 1098 - 52)

The committee withdrew from the preprinted report the recommendations for the revision of Standard Method of Test for Moisture in Paper, Paperboard, and Paperboard and Fiberboard Containers (D 644 - 55), the reapproval of Standard Methods D 527 - 41, D 589 - 44, D 643 - 43, D 722 - 45, D 724 - 45, D 725 - 55, D 776 - 46, D 825 - 54, D 826 - 47, D 827 - 47, D 829 - 48 and D 722 - 45, the continuation without revision of Tentative Methods D 987 - 48 T, and the editorial change relating to all chemical methods containing a section on Reagents.

Committee D-14 on Adhesives:

Report presented in the absence of the chairman by S. F. Etris, ASTM Staff, and the following actions taken:

Accepted as Tentative:

Test for Permanence of Adhesive-Bonded Joints in Plywood Under Mold Conditions (D 1877 - 61 T)

Test for Peel Resistance of Adhesives (T-Peel Test) (D 1876 - 61 T)

Test for Density of Adhesives in Fluid Form (D 1875 - 61 T)

Test for Pressure-Sensitive Tack of Adhesives (D 1878 - 61 T)

Spec. for Water- or Solvent-Soluble Liquid Adhesives for Automatic Machine Sealing of Top Flaps of Fiberboard Shipping Cases (D 1874 - 61 T)

Rec. Practice for Exposure of Adhesive Specimens to High-Energy Radiation (D 1879 - 61 T)

Accepted as Tentative, Revisions in:

Tests for Resistance of Adhesives for Wood to Cyclic Laboratory Aging Conditions (D 1183 - 55 T)
Def. of Terms Relating to Adhesives (D 907 - 60)

Adopted as Standard:

Test for Measuring the Hydrogen Ion Concentration of Dry Adhesive Film (D 1583 - 58 T)
Test for Effect of Moisture and Temperature on Adhesive Bonds (D 1151 - 58 T)

Committee D-16 on Industrial Aromatic Hydrocarbons and Related Materials:

Report presented by W. E. Sisco, chairman, and the following actions taken:

Accepted for Publication as Information Only:

Proposed Test for Aldehydes in Styrene Monomer
Proposed Test for Solubility of Styrene Polymer
Proposed Test for Inhibitor, 4-*Tert*-Butylcatechol in Styrene Monomer
Proposed Test for Polymer Content of Styrene Monomer

Accepted as Tentative, Revisions in:

Test for Traces of Thiophene in Benzene Using Isatin and Spectrophotometry (D 1685 - 59 T)

Adopted as Standard:

Test for Color of Solid Aromatic Hydrocarbons and Related Materials in the Molten State (Platinum-Cobalt Scale) (D 1686 - 59 T)

Committee D-20 on Plastics:

Report presented by J. B. DeCoste, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Styrene-Butadiene Molding and Extrusion Materials (D 1892 - 61 T)
Test for Blocking of Plastic Film (D 1893 - 61 T)
Test for Coefficients of Friction of Plastic Film (D 1894 - 61 T)
Test for Apparent Density, Bulk Factor, and Pourability of Plastic Materials (D 1895 - 61 T)
Rec. Practice for Transfer Molding of Thermosetting Materials (D 1896 - 61 T)
Rec. Practice for Injection Molding of Specimens of Polystyrene Molding and Extrusion Materials (D 1897 - 61 T)
Rec. Practice for Sampling of Plastics (D 1898 - 61 T)
Test for Elevated Temperature Aging Using a Tubular Oven (D 1870 - 61 T). (This test was prepared jointly by Committees D-11 on Rubber and D-20 on Plastics and was appended to the D-11 Report. While the D-20 Report carried no recommendation for its publication as tentative, Committee D-20 has approved the recommendation.)
Methods of Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (D 392 - 38) (Standard reverted to tentative without change)
Test for Compressive Properties of Rigid Plastics (D 695 - 54) (Standard reverted to tentative without change)
Test for Luminous Reflectance, Transmittance, and Color of Materials (D 791 - 54) (Standard reverted to tentative without change)

Accepted as Tentative, Revisions in:

Spec. for Cellulose Acetate Molding and Extrusion Compounds (D 706 - 55 T)
Spec. for Cellulose Acetate Butyrate Molding and Extrusion Compounds (D 707 - 55 T)
Spec. for Nylon Injection Molding and Extrusion Materials (D 789 - 59 T)
Test for Stiffness in Flexure of Plastics (D 747 - 58 T)
Methods of Testing Urethane Foam Raw Materials (D 1638 - 60 T)
Def. of Terms Relating to Plastics (D 883 - 60 T)
Spec. for Molds for Test Specimens of Plastic Molding Materials (D 647 - 57)
Test for Stiffness Properties of Nonrigid Plastics as a Function of Temperature by Means of a Torsional Test (D 1043 - 51) (Standard reverted to tentative)
Rec. Practice for Molding Test Specimens of Phenolic Materials (D 796 - 51) (Standard reverted to tentative)

Adopted as Standard:

Test for Flammability of Plastics 0.050 in. and Under in Thickness (D 568 - 56 T)
Test for Flexural Properties of Plastics (D 790 - 59 T)
Test for Ammonia in Phenol-Formaldehyde Molded Materials (D 834 - 59 T)
Test for Haze and Luminous Transmittance of Transparent Plastics (D 1003 - 59 T)
Test for Tear Resistance of Plastic Film and Sheeting (D 1004 - 59 T)
Test for Orientation Release Stress of Plastic Sheeting (D 1504 - 57 T)
Test for Dilute Solution Viscosity of Ethylene Polymers (D 1601 - 59 T)
Test for Tensile Heat Distortion Temperature of Plastic Sheeting (D 1637 - 59 T)

Test for Indentation Hardness of Plastics by Means of a Durometer (D 1706 - 59 T)
Test for Particle Size Analysis of Powdered Polymers and Copolymers of Vinyl Chloride (D 1705 - 59 T)
Spec. for Extruded Acrylic Plastic Sheet (D 1547 - 59 T)
Spec. for Nonrigid Vinyl Chloride Plastic Sheeting (D 1593 - 58 T)

Adopted as Standard, Revisions in:

Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 - 58) (Jointly with Committee D-9) covering (1) adoption as standard of tentative revisions issued in 1959; and (2) immediate adoption of revisions of Section 3(*d*)

Standard Withdrawn:

Method for Estimating Blocking of Plastic Sheets (D 884 - 48)

The committee withdrew from the preprinted report the recommendation for adoption as standard of Tentative Method of Test for Brittleness Temperature of Plastics and Elastomers by Impact (D 746 - 57 T)

Committee D-25 on Casein and Similar Protein Materials:

Report presented in the absence of the chairman by S. F. Etris, ASTM Staff, and accepted as a report of progress.

Committee E-10 on Radioisotopes and Radiation Effects:

Report presented in the absence of the chairman by G. W. Pomeroy, vice-chairman, and the following actions taken:

Accepted as Tentative:

General Methods for Analysis of Radioisotopes (E 181 - 61 T)
Methods for Analysis of Phosphorus-32 (E 182 - 61 T)
Rec. Practice for Determining Changes in Chemical Reactivity of Inorganic Materials Exposed to High-Energy Radiation (E 183 - 61 T)
Rec. Practice for Effect of High-Energy Radiation on the Tensile and Impact Properties of Metallic Materials (E 184 - 61 T)
Rec. Practice for Surveillance Tests on Structural Materials in Nuclear Reactors (E 185-61 T)

Committee F-2 on Flexible Barrier Materials:

Report presented in the absence of the chairman by S. F. Etris, ASTM Staff, and the following action taken:

Accepted as Tentative:

Def. of Terms Relating to Flexible Barrier Materials (F 17 - 61 T)

THIRTY-SIXTH SESSION—SESSION ON CEMENT

THURSDAY, JUNE 29, 8:00 P.M.

SESSION CHAIRMAN: W. S. WEAVER

Investigation of Concrete Materials for a Major Project in Western Canada—G. C. Price, presented by the author.

Improved Adiabatic Calorimeter for Concrete—David Pirtz, presented by the author.[25]

The Mechanism of Grinding Aids—F. J. Mardulier, presented by the author.

Significance of Selected ASTM C-1 Tests:

Discussion of Tentative Methods of Test for False Set of Portland Cement—W. C. Hansen, presented by the author.
Significance of Test for Calcium Sulfate in Hydrated Portland Cement Mortar—T. B. Kennedy, presented by the author.
Significance of Tests for Sulfate Resistance—William Lerch, presented by the author.
Flexural Strength of Hydraulic Cement Mortars—M. A. Swayze, presented by J. L. Sawyer.

PANEL DISCUSSION ON DISTRICT ACTIVITIES

THURSDAY, JUNE 29, 8:00 P.M.

The Administrative Committee on District Activities sponsored an open panel discussion on district activities. The topics covered included discussion of revised district charter and manual, programs for districts, and student award programs. All officers and members of district councils and all others interested in district affairs were invited to attend. This program is part of the effort of ACDA to stimulate greater and more effective district activities.

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[25] Published in *Materials Research & Standards*, Vol. 2, No. 1, Jan., 1962.

THIRTY-SEVENTH SESSION—SESSION ON ROAD AND PAVING MATERIALS[26]

THURSDAY, JUNE 29, 8:00 P.M.

SESSION CHAIRMAN: J. O. IZATT

A Laboratory-Field Study of Hot Asphaltic Concrete Wearing Course Mixtures—J. F. Goode, presented by the author.

Significance of Variation of Bitumen Content of Paving Mixtures—J. H. Keyser and N. G. Gaudette, presented by Mr. Keyser.

Immediate Core Volume by Presaturation—L. E. Santucci and R. J. Schmidt, presented by R. S. Winniford.

Air Permeability of Asphalt Concrete—T. C. Hein and R. J. Schmidt, presented by R. S. Winniford.

Measurement of Asphalt Viscosity with a Vacuum Capillary Viscometer—J. J. Heithaus, presented by the author.

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[26] The Session on Road and Paving Materials and the Symposium on Microviscometry were published as Papers on Road and Paving Materials and Symposium on Microviscometry, *ASTM STP No. 309.*

THIRTY-EIGHTH SESSION—SYMPOSIUM ON IMPURITIES IN STEAM

THURSDAY, JUNE 29, 8:00 P.M.

SESSION CHAIRMAN: J. K. RICE

Introduction by J. K. Rice.

The Stoichiometry of the Vaporous Carry-over of Sodium Chloride from High-Pressure Boiler Water (ASTM-Sponsored Research Report)—M. M. Rubright, presented by the author.

The Prevention of Errors in Steam Purity Measurement Caused by Deposition of Impurities in Sampling Lines—R. V. Cobb and E. E. Coulter, presented by Mr. Cobb.

Impurities in Steam from High-Pressure Boilers —R. C. Ulmer and H. A. Klein, presented by Mr. Klein.

Summary by F. E. Clarke.

PAPERS—PANEL DISCUSSION ON TRANSIENT TEMPERATURE MEASUREMENTS WITH THERMOCOUPLES IN MISSILE AND SPACE APPLICATION

THURSDAY, JUNE 29, 8:00 P.M.

The following papers were presented at an informal session sponsored by Subcommittee 32 on Thermocouples for Temperature Measurement of Committee E 1 on Methods of Testing.

Temperature Determination on the Pershing Missile—D. Booker, P. Moran, and R. Ahearn.

Spring-Loaded Thermocouple Probes and Transient Temperatures—P. M. Hahn.[27]

Panel Discussion:

Panel Members: J. W. Freeman, University of Michigan.
A. I. Dahl, General Electric Co.
E. H. Evans, Boeing Co.

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[27] To be published in *Materials Research & Standards.*

THIRTY-NINTH SESSION—SYMPOSIUM ON MICROVISCOMETRY[26]

FRIDAY, JUNE 30, 9:30 A.M.

SESSION CHAIRMAN: A. B. CORNTHWAITE

Measurement of Consistency of Paving Cements at 140 F with the Sliding Plate Microviscometer—D. F. Fink and R. L. Griffin, presented by Mr. Fink.

The Precision of Measurements with the Sliding Plate Microviscometer—D. F. Fink and J. J. Heithaus, presented by Mr. Heithaus.

The Rheology of Asphalt-Filler Systems as Shown by the Microviscometer—R. S. Winniford, presented by the author.

The Effects of Viscosity in Hot Mix Construction—Verdi Adam, presented by J. O. Izatt.

Changes in Asphalt Viscosities During the Thin-Film Oven and Microfilm Durability Tests—W. J. Halstead and J. A. Zenewitz, presented by Mr. Halstead.

FORTIETH SESSION—COMMITTEE REPORT SESSION

FRIDAY, JUNE 30, 12:30 P.M.

SESSION CHAIRMAN: J. B. RATHER

Committee D-2 on Petroleum Products and Lubricants:

Report presented by H. M. Smith, chairman, and the following actions taken:

Accepted for Publication as Information Only:

Proposed Method of Test for Separation of Tetraethyllead and Tetramethyllead in Gasoline
Proposed Method of Test for Residues in Liquefied Petroleum (LP) Gases (End Point Index Method)
Proposed List of Methods for Use in Specifications of Commercial Butane-Butene Mixtures
Proposed Method of Test for Knock Characteristics of Motor Fuels by Research Method Equipped with a Split-Head Cylinder Engine
Proposed Method of Sampling Turbine Engine Fuel for Cleanliness Evaluation
Proposed Method of Test for Barium, Calcium, and Zinc in Lubricating Oils and Additives (Complexometric Titration)
Proposed Method of Test for Hydrocarbon Types in Low-Olefinic Gasolines by Mass Spectroscopy
Proposed Method of Test for Hydrocarbon Types in Olefinic Gasolines by Mass Spectrometry
Proposed Centrifuge Method for Preparing Specimen Grids for Use in the Study of Diesel Fuels by Electron Microscopy
Proposed Method of Test for Solid Point of Aviation Turbine Fuels
Proposed Method of Test for Vapor Pressure of Petroleum Products (Micro Method)
Proposed Method of Test for Shear Stability of Polymer-Thickened Oils
Proposed Method of Test for Knock Characteristics of Motor Fuels: by the 1170 Severity Method

Accepted as Tentative:

Spec. for Commercial Hexanes (D 1836 – 61 T)
Specs. for Liquefied Petroleum (LP) Gases (D 1835 – 61 T)
Method of Test for Volatility of Liquefied Petroleum (LP) Gases (D 1837 – 61 T)
Test for Copper Strip Corrosion by Liquefied Petroleum (LP) Gases (D 1838 – 61 T)
Test for Roll Stability of Lubricating Grease (D 1831 – 61 T)
Test for Peroxide Number of Petroleum Wax (Peroxide Method) (D 1832 – 61 T)
Test for Odor of Petroleum Wax (D 1833 – 61 T)
Test for 20-deg Specular Gloss of Waxed Paper (D 1834 – 61 T)
Test for Amyl Nitrate in Diesel Fuels (D 1839 – 61 T)
Test for Naphthalene Hydrocarbons in Aviation Turbine Fuels by Ultraviolet Spectrophotometry (D 1840 – 61 T)
Test for Oxygen in Light Hydrocarbon Vapors (D 1021 – 55) (Standard reverted to tentative without change)

The committee submitted for publication as information the following five methods which did not appear in the report as preprinted:

Proposed Method of Analysis of Natural Gas by Gas Chromatography
Proposed Rapid Method of Test for Oxidation Stability of Steam Turbine Oils
Proposed Method for Accelerated Stability Test for Distillate Fuel Oil
Proposed Method of Test for Particulate Matter in Aviation Turbine Fuels
Three Proposed Methods for Analysis of Rubber Extender Oils and Results of Cross-Test Program (Comparison of Methods by Joint ASTM Committees D-2 and D-11 Subcommittee on Rubber Extender and Process Oils):
  Method I. Chromatographic Analysis of Petroleum Oils
  Method II. Modified Rostler-Sternberg Method for Composition of Petroleum Oils
  Method III. Clay-Gel Adsorption Chromatographic Method for Characteristic Groups in Rubber Extender Oils

Accepted as Tentative, Revisions in:

Test for Existent Gum in Fuels by Jet Evaporation (D 381 – 58 T)
Spec. for Fuel Oils (D 396 – 60 T)
Test for Unsulfonated Residue of Petroleum Plant Spray Oils (D 483 – 60 T)
Test for Ignition Quality of Diesel Fuels by the Cetane Method (D 613 – 59 T)
Test for Hydrocarbon Types in Liquid Petroleum Products by Fluorescent Indicator Adsorption (D 1319 – 60 T)
Test for Needle Penetration of Petroleum Waxes (D 1321 – 60 T)
Test for Estimation of Net Heat of Combustion of Liquid Petroleum Products (D 1405 – 57 T)
Spec. for Aviation Turbine Fuels (D 1655 – 59 T)

Test for Knock Characteristics of Motor Fuels Above 100 Octane Number by the Research Method (D 1656 – 60 T)
Test for Thermal Stability of Aviation Turbine Fuels (D 1660 – 60 T)
Method for Analysis of Commercial Butane-Butene Mixtures (Gas Chromatography) (D 1717 – 60 T)

Adopted as Standard:

Test for Flash Point by Pensky-Martens Closed Tester (D 93 – 58 T)
Test for Bromine Number of Petroleum Distillates by Electrometric Titration (D 1159 – 59 T)
Test for Distillation at Reduced Pressure of Petroleum Products (D 1160 – 57 T)
Methods for Measurement of Refractive Index and Refractive Dispersion of Hydrocarbon Liquids (D 1218 – 52 T)
Test for Leakage Tendencies of Automotive Wheel Bearing Greases (D 1263 – 53 T)
Method of Sampling Petroleum and Petroleum Products (D 270 - 55 T, as revised

Adopted as Standard, Revisions in:

Test for Flash Point by Tag Closed Tester (D 56 – 56) (Jointly with Committee D-1)
Test for Distillation of Petroleum Products (D 86 – 59)
Test for Precipitation Number of Lubricating Oils (D 91 – 52)
Test for Water in Petroleum Products and Other Bituminous Materials (D 95 – 58) (Approved subject to the concurrence of Committee D-4, which committee has joint jurisdiction)[28]
Methods of Analysis of Lubricating Grease (D 128 – 59)
Test for Conradson Carbon Residue of Petroleum Products (D 189 – 58)
Def. of Terms Relating to Petroleum (D 288 – 57)
Test for Knock Characteristics of Motor Fuels Below 100 Octane Number by the Motor Method (D 357 – 60)
Test for Kinematic Viscosity (D 445 – 60)
Test for Tetraethyllead in Gasoline (D 526 – 56)
Test for Knock Characteristics of Motor Fuels Below 100 Octane Number by the Research Method (D 908 – 60)
Test for Alpha Acetylenes in Light Hydrocarbons (D 1020 – 59)
Test for Mercaptan Sulfur in Aviation Turbine Fuels (Color-Indicator Method) (D 1219 – 56)
Test for Tetraethyllead in Gasoline (Polarographic Method) (D 1269 – 58)
Test for Mercaptan Sulfur in Aviation Turbine Fuels (Amperometric Method) (D 1323–56)

Standard Withdrawn:

Method of Analysis of 60 Octane Number *Iso*-octane - Normal Heptane ASTM Knock Test Reference Fuel Blends by Infrared Spectrophotometry (D 1095 – 54)

The committee withdrew from the preprinted report the recommendations for revision of Tentative Method of Test for Luminometer Numbers of Aviation Turbine Fuels (D 1740 – 60 T),[29] and the withdrawal of Standard Method of Test for Saponification Number by Potentiometric Titration (D 939 – 54)

Committee D-4 on Road and Paving Materials:

Report presented by R. E. Bollen, chairman, and the following actions taken:

Accepted as Tentative:

Test for Recovery of Asphalt from Solution by Abson Method (D 1856 – 61 T)
Methods of Testing Concrete Joint Sealers, Cold-Application Type (D 1851 – 61 T)
Methods of Testing Jet-Fuel-Resistant Joint Sealer for Concrete, Cold-Application Elastic Type (D 1853 – 61 T)
Methods of Testing Jet-Fuel-Resistant Concrete Joint Sealer, Hot-Poured Elastic Type (D 1855 – 61 T)
Specs. for Concrete Joint Sealer, Cold-Application Type (D 1850 – 61 T)
Specs. for Jet-Fuel-Resistant Concrete Joint Sealer, Cold-Application Elastic Type (D 1852 – 61 T)
Specs. for Jet-Fuel-Resistant Concrete Joint Sealer, Hot-Poured Elastic Type (D 1854 – 61 T)

Accepted as Tentative, Revisions in:

Spec. for Crushed Stone, Crushed Slag, and Gravel for Single or Multiple Bituminous Surface Treatments (D 1139 – 57 T)
Methods of Testing Emulsified Asphalts (D 244 – 60)
Test for Amount of Material Finer than No. 200 Sieve in Aggregate (C 117 – 49) (Standard reverted to tentative) (Jointly with Committee C-9)

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[28] There has been no opportunity for this recommendation to receive consideration in Committee D-4 and accordingly it does not appear on the Society letter ballot. This proposed revision, however, will be published as information in an Editorial Note at the end of Method D 95.—ED.

[29] This proposed revision will be published as information in an Editorial Note at the end of Method D 1740.—ED.

Test for Sieve Analysis of Fine and Coarse Aggregates (C 136 – 46) (Standard reverted to tentative) (Jointly with Committee C-9)

Spec. for Bituminous Mixing Plant Requirements (D 995 – 55) (Standard reverted to tentative)

Adopted as Standard:

Test for Vacuum Distillation of Liquid and Semi-Solid Asphaltic Materials to Obtain a Residue of Specified Penetration (D 1189 – 52 T)

Test for Engler Specific Viscosity of Tar Products (D 1665 – 59 T)

Test for Penetration of Bituminous Materials (D 5 – 59 T)

Standard Withdrawn:

Test for Penetration of Bituminous Materials (D 5 – 52)

Committee D-9 on Electrical Insulating Materials:

Report presented in the absence of the chairman by L. J. Timm, and the following actions taken:

Accepted as Tentative:

Method for Corona Measurement (D 1868 – 61 T)

Specs. for Copper-Clad Thermosetting Laminates for Printed Wiring (D 1867 – 61 T) with the addition to Table I of requirements for water absorption for thicknesses of $\frac{1}{32}$ in., $\frac{1}{16}$ in., $\frac{3}{32}$ in. and $\frac{1}{8}$ in.

Accepted as Tentative, Revisions in:

Spec. for Vulcanized Fibre Sheets, Rods, and Tubes Used for Electrical Insulation (D 710 – 54 T)

Methods of Testing Vitrified Ceramic Materials for Electrical Applications (D 116 – 60 T)

Methods of Testing Sheet and Plate Materials Used for Electrical Insulation (D 229 – 58) (Standard reverted to tentative)

Adopted as Standard:

Spec. for Orange Shellac and Other Indian Lacs for Electrical Insulation (D 784 – 59 T)

Spec. for Absorbent Laminating Paper for Electrical Insulation (D 1080 – 58 T)

Test for High-Voltage, Low-Current Arc Resistance of Solid Electrical Insulating Materials (D 495 – 58 T)

Methods of Testing Nonrigid Vinyl Chloride Polymer Tubing (D 876 – 60 T)

Methods of Testing Pressure-Sensitive Adhesive Coated Tapes Used for Electrical Insulation (D 1000 – 61 T)

Methods of Testing Hydrocarbon Waxes Used for Electrical Insulation (D 1168 – 58 T)

Test for Dielectric Constant and Dissipation Factor of Polyethylene by Liquid Displacement Procedure (D 1531 – 59 T)

Test for Dielectric Constant and Dissipation Factor of Expanded Cellular Plastics Used for Electrical Insulation (D 1673 – 59 T)

Methods of Testing Polymerizable Embedding Compounds Used for Electrical Insulation (D 1674 – 59 T)

Adopted as Standard, Revisions in:

Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 – 58) (Jointly with Committee D-20) covering (1) adoption as standard of tentative revisions issued in 1959; and (2) immediate adoption of revisions of Section 3(*d*)

Test for Electrical Resistance of Insulating Materials (D 257 – 58)

Test for Dielectric Breakdown Voltage and Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (D 149 – 59)

Methods of Testing Varnished Cotton Fabrics and Varnished Cotton Fabric Tapes Used for Electrical Insulation (D 295 – 58)

Methods of Testing Varnished Glass Fabrics and Varnished Glass Fabric Tapes Used for Electrical Insulation (D 902 – 56)

Spec. for Nonrigid Vinyl Plastic Tubing (D 922 – 59)

Committee D-11 on Rubber and Rubber-like Materials:

Report presented by Simon Collier, chairman, and the following actions taken:

Accepted as Tentative:

Specs. for Rubber Rings for Asbestos-Cement Pipe (D 1869 – 61 T)

Test for Elevated Temperature Aging Using a Tubular Oven (D 1870 – 61 T) (Jointly with Committee D-20) (A number of editorial changes were made in this method.)[30]

Test for Adhesion of Vulcanized Rubber to Single-Strand Wire (D 1871 – 61 T)

Test for Free 2-Mercaptobenzothiazole in Benzothiazyl Disulfide Rubber Vulcanization Accelerator (D 1872 – 61 T)

Test for Total 2-Mercaptobenzothiazole in Commercial Benzothiazyl Disulfide Rubber Vulcanization Accelerator (D 1873 – 61 T)

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[30] It was recommended that this method be referred to Committee E-1 on Methods of Testing for review, particularly of the oven requirements before publication.—Ed.

Accepted as Tentative, Revisions in:

Methods for Chemical Analysis of Rubber Products (D 297 - 60 T)
Spec. for Elastomer Compounds for Automotive Applications (D 735 - 60 T)
Test for Compressibility and Recovery of Gasket Materials (D 1147 - 59 T)
Methods for Chemical Analysis of Synthetic Elastomers (Solid Styrene-Butadiene Copolymers) (D 1416 - 60 T)
Rec. Practice for Nomenclature for Synthetic Elastomers and Latices (D 1418 - 58 T)
Rec. Practice for Description of Types of Styrene-Butadiene Rubbers (SBR) and Butadiene Rubbers (BR) (D 1419 - 60a T)
Spec. for Carbon Blacks Used in Rubber Products (D 1765 - 60 T) (Jointly with Committee D-24)

Adopted as Standard:

Method of Measuring Low-Temperature Stiffening of Rubber and Rubber-like Materials by Means of a Torsional Wire Apparatus (D 1053 - 58 T), as revised
Test for Viscosity and Curing Characteristics of Rubber by the Shearing Disk Viscometer (D 1646 - 59 T), as revised
Test for Abrasion Resistance of Rubber Soles and Heels (D 1630 - 59 T)
Methods of Testing Adhesives for Brake Linings and Other Friction Materials (D 1205 - 59 T)
Method for Strain Testing of Vulcanized Rubber (D 1456 - 57 T)

Adopted as Standard, Revisions in:

Test for Compression Set of Vulcanized Rubber (D 395 - 55)
Test for Resistance to Surface Cracking of Stretched Rubber Compounds (D 518 - 60)
Test for Accelerated Aging of Vulcanized Rubber by the Oxygen-Pressure Method (D 572 - 53)
Test for Physical State of Cure of Vulcanized Rubber (T-50 Test) (D 599 - 55)
Test for Weather Resistance Exposure of Automotive Rubber Compounds (D 1171 - 59)
Methods of Testing Synthetic Rubber Latices (Styrene-Butadiene Copolymers) (D 1417 - 59)

The committee withdrew from the preprinted report the recommendations for revision of Tentative Methods for Chemical Analysis of Natural Rubber (D 1278 - 61 T), and the adoption as standard without revision of Tentative Methods of Test D 471 - 59 T, D 1390 - 56 T, and D 1415 - 56 T.

Committee D-15 on Engine Antifreezes:

Report presented by R. E. Vogel, chairman, and the following actions taken:

Accepted as Tentative:

Method of Glassware Test for Foaming Tendencies of Engine Antifreezes (D 1881 - 61 T)
Test for Effect of Antifreeze Solutions on Organic Finishes for Automotive Vehicles (D 1882 - 61 T)
Rec. Practice for Selection of Engine Antifreezes for Use in Automotive Cooling Systems, Ethylene Glycol and Methanol Types (D 1880 - 61 T)

Accepted as Tentative, Revisions in:

Method for Glassware Corrosion Test for Engine Antifreezes (D 1384 - 55 T)

Adopted as Standard:

Spec. for Hydrometer-Thermometer Field Tester for Engine Antifreezes (D 1124 - 58 T)

Committee D-19 on Industrial Water:

Report presented in the absence of the chairman by P. J. Smith, ASTM Staff, and the following actions taken subject to a favorable vote of the committee on the report:

Accepted as Tentative:

Test for Acidity and Alkalinity of Industrial Water (D 1884 - 61 T)
Test for Chloride Ion in High-Purity Industrial Water (D 1885 - 61 T)
Test for Nickel in Industrial Water (D 1886 - 61 T)
Test for Suspended and Dissolved Solids in Industrial Water (D 1888 - 61 T)
Test for Sodium in High-Purity Industrial Water by Flame Photometry (D 1887 - 61 T)
Test for Turbidity of Industrial Water (D 1889 - 61 T)
Test for Hexane-Extractable Matter in Industrial Waste Water (D 1891 - 61 T)
Method for Measurement of Beta Particle Radioactivity of Industrial Water and Industrial Waste Water (D 1890 - 61 T)

Accepted as Tentative, Revisions in:

Tests for Iron in Industrial Water and Industrial Waste Water (D 1068 - 60 T)
Test for Silica in High-Purity Water (D 1689 - 59 T)
Test for Odor in Industrial Water and Industrial Waste Water (D 1292 - 59 T)

Adopted as Standard:

Scheme for Analysis of Industrial Water and Industrial Waste Water (D 1256 - 59 T)
Tests for Sodium and Potassium Ions in Industrial Water and Water-Formed Deposits by

Flame Photometry (D 1428 - 56 T), with the deletion of the proposed Sections 13(*b*) and 19(*b*).

Tests for Total Chromium in Industrial Water and Industrial Waste Water (D 1687 - 59 T)

Method for Measurement of Gamma Radioactivity of Industrial Water and Industrial Waste Water (D 1690 - 59 T)

Adopted as Standard, Revisions in:

Def. of Terms Relating to Industrial Water and Industrial Waste Water (D 1129 - 60)

Tests for Fluoride Ion in Industrial Water and Industrial Waste Water (D 1179 - 58)

Tentative Withdrawn:

Tests for Acidity and Basicity (Alkalinity) in Industrial Water and Industrial Waste Water (D 1067 - 57 T)

Standard Withdrawn:

Tests for Suspended and Dissolved Matter (Suspended and Dissolved Solids) in Industrial Water and Industrial Waste Water (D 1069 - 58)

Committee D-26 on Halogenated Organic Solvents:

Report presented by W. D. McMaster, chairman, and the following action taken:

Accepted as Tentative:

Test for Relative Evaporation Time of Halogenated Hydrocarbons and Their Admixtures (D 1901 - 61 T)

Committee D-27 on Electrical Insulating Liquids and Gases:

Report presented in the absence of the chairman by C. A. Johnson, secretary, and the following actions taken:

Accepted as Tentative:

Test for Approximate Acidity and Polar Contamination in Used Mineral Transformer Oil by Spot Tests (D 1902 - 61 T)

Test for Coefficient of Thermal Expansion of Electrical Insulating Liquids of Petroleum Origin, and Askarels (D 1903 - 61 T)

Test for Oxidation Characteristics of Mineral Transformer Oils (D 1904 - 61 T)

Accepted as Tentative, Revisions in:

Test for Approximate Acidity of Used Electrical Insulating Oil of Petroleum Origin (D 1534 - 58 T)

Test for 2,6 Ditertiary-Butyl Para-Cresol in New Electrical Insulating Oils (D 1473 - 60) (Standard reverted to tentative)

Adopted as Standard, Revisions in:

Test for Water in Insulating Liquids (Karl Fischer Method) (D 1533 - 60)

Tentative Withdrawn:

Test for Free Chlorides in Askarels (D 1811 - 60 T)

FORTY-FIRST SESSION—COMMITTEE REPORT SESSION

FRIDAY, JUNE 30, 12:30 P.M.

SESSION CHAIRMAN: E. C. SHUMAN

Committee C-1 on Cement:

Report presented in the absence of the chairman by W. S. Weaver, vice-chairman, and the following actions taken:

Accepted as Tentative:

Specs. for Processing Additions for Use in the Manufacture of Portland Cement (C 465 - 61 T)

Accepted as Tentative, Revisions in:

Methods of Chemical Analysis of Portland Cement (C 114 - 58 T)

Methods of Sampling Hydraulic Cement (C 183 - 60 T)

Specs. for Portland Blast-Furnace Slag Cement (C 205 - 60 T)

Specs. for Air-Entraining Additions for Use in the Manufacture of Air-Entraining Portland Cement (C 226 - 58 T)

Specs. for Flow Table for Use in Tests of Hydraulic Cement (C 230 - 57 T)

Test for Flexural Strength of Hydraulic Cement Mortars (C 348 - 57 T)

Test for False Set of Portland Cement (Mortar Method) (C 359 - 56 T)

Spec. for Portland Cement (C 150 - 60)

Adopted as Standard, Revisions in:

Specs. for Portland Cement (C 150 - 60): covering (1) adoption as standard of tentative revision of Section 4; and (2) immediate adoption of revisions of Section 3 and Tables I and II

Specs. for Air-Entraining Portland Cement (C 175 - 60): covering (1) adoption as standard of tentative revision of Section 4; and (2) immediate adoption of revisions of Tables I and II

Methods of Chemical Analysis of Portland Cement (C 114 - 58)

Committee C-11 on Gypsum:

Report presented in the absence of the chairman by H. Omson, secretary, and the following actions taken:

Accepted as Tentative:

Specs. for Joint Treatment Materials for Gypsum Wallboard Construction (C 475 - 61 T)

Methods of Testing Joint Treatment Materials for Gypsum Wallboard Construction (C 474 - 61 T)

Accepted as Tentative, Revisions in:

Spec. for Annular Ringed Nails for Gypsum Wallboard (C 380 - 60 T)

Spec. for Gypsum Backing Board (C 442 - 59 T)

Adopted as Standard:

Methods of Chemical Analysis of Gypsum and Gypsum Products (C 471 - 61 T)

Methods of Physical Testing of Gypsum Plasters and Gypsum Concrete (C 472 - 61 T)

Methods of Physical Testing of Gypsum Board Products and Gypsum Partition Tile or Block (C 473 - 61 T)

Adopted as Standard, Revisions in:

Spec. for Gypsum Wallboard (C 36 - 60)

Standard Withdrawn:

Methods of Testing Gypsum and Gypsum Products (C 26 - 60)

Committee E-2 on Emission Spectroscopy:

Report presented by R. E. Michaelis, chairman, and the following actions taken:

Accepted for Publication as Information Only:

Suggested Method for Spectrochemical Analysis of Indium-Gallium Alloys by the Solution-Graphite Spark Technique

Suggested Method for Spectrochemical Analysis of Blast Furnace and Steelmaking Slags by the Fusion Spark Technique

Adopted as Standard:

Method for Spectrochemical Analysis of Nickel Alloys by the Powder—D-C Arc Technique (E 129 - 57 T)

Committee E-5 on Fire Tests of Materials and Construction:

Report presented in the absence of the chairman by I. A. Benjamin, secretary, and the following actions taken:

Accepted as Tentative:

Def. of Terms Relating to Fire Tests of Building Construction and Materials (E 176 - 61 T)

Accepted as Tentative, Revisions in:

Methods of Fire Tests of Building Construction and Materials (E 119 - 58)

Adopted as Standard:

Test for Surface Burning Characteristics of Building Materials (E 84 - 60 T)

Adopted as Standard, Revisions in:

Methods of Fire Tests of Building Construction and Materials (E 119 - 58): covering (1) adoption as standard of tentative revision of Sections 9 and 30; and (2) immediate adoption of revisions of Sections 4 and 9(*c*), and the addition of Appendix II.

Committee E-7 on Nondestructive Testing:

Report presented by J. H. Bly, chairman, and accepted as a report of progress.

Committee E-13 on Absorption Spectroscopy:

Report presented in the absence of the chairman by R. F. Robey, secretary, and the following actions taken:

Accepted for Publication as Information Only:

Proposed Rec. Practices for Identification of Materials by Absorption Spectroscopy, Using the Wyandotte - ASTM (Kuentzel) Punched Card Index

Accepted as Tentative, Revisions in:

Def. of Terms and Symbols Relating to Absorption Spectroscopy (E 131 - 59 T)

Committee E-15 on Analysis and Testing of Industrial Chemicals:

Report presented by W. A. Kirklin, chairman, and the following action taken:

Accepted as Tentative:

Rec. Practice for Developing Precision Data on ASTM Methods for Analysis and Testing of Industrial Chemicals (E 180 - 61 T)

Committee E-19 on Gas Chromatography:

Report presented in the absence of the chairman by L. C. Gilbert, ASTM Staff, and accepted as a report of progress subject to letter ballot approval by the committee.

Joint Committee on Leather:

Report presented in the absence of the chairman by Bruce L. Lewis, vice-chairman, and the following action taken:

Accepted as Tentative:

Test for Cold-Crack Resistance of Upholstery Leather (D 1912 – 61 T)

EDITOR'S NOTE.—The recommendations affecting standards referred to letter ballot were approved when the ballot was canvassed on September 18.

SUMMARY OF PROCEEDINGS OF CINCINNATI, OHIO SPRING MEETING

During the 1961 ASTM Committee Week held in Cincinnati, Ohio, a Symposium on Fire Test Methods was held on Wednesday, Feb. 1, under the sponsorship of Committee E-5 on Fire Tests of Materials and Construction, as follows:

SYMPOSIUM ON FIRE TEST METHODS

Fire tests of materials used in building construction are of very serious concern to those responsible for establishing criteria for qualifying these materials for use in buildings, for the protection and safety of human life are involved. Therefore fire test methods are scrutinized very carefully as to whether they simulate properly the actual behavior of materials when exposed to fire conditions.

The following papers and discussion are included in the symposium:

Early History of Fire Endurance Testing in the United States—Harry Shoub

A Treatise on Theoretical Fire Testing—T. Z. Harmathy

The New Beam Furnace at PCA and Some Experience Gained from Its Use—C. C. Carlson and Phil J. Tatman

Surface Flammability of Materials: A Study of Test Methods and Comparison of Results—J. A. Wilson

These papers with discussion were issued as *ASTM Special Technical Publication No. 301*, entitled "Symposium on Fire Test Methods."

AN ASTM FOR THE FUTURE*

ANNUAL ADDRESS BY THE PRESIDENT

A. ALLAN BATES[1]

Fellow members of ASTM, this 60th year of our existence under charter as the American Society for Testing Materials finds us in mid-passage between two historical periods so profoundly different one from the other that they cannot be thought of only as "past" and "future." For those of us concerned with materials an "old world" is closing and a "new world" is opening. The future is not to be simply a projection and enlargement of the past. Just as the voyages of Columbus, Magellan, da Gama, and Cabot all in one generation revealed horizons of the earth previously unbelievable, so have explorations among molecules, atoms, and nuclei in our present scientific laboratories opened realms inconceivable in the earlier working years of many present ASTM members.

We all sense the impending great change, dimly. None can foresee its full portents and probably few will quickly appreciate its deeper possibilities for human enrichment. Ferdinand and Isabella bade their voyagers to bring back gold, gold, and more gold, and thus they lost the new world and its earthly wealth a millionfold greater than its gold.

ASTM HAS DONE WELL IN THE PAST

Since its formal birth in March of 1902, the American Society for Testing Materials has uniquely and magnificently served a purpose without which industrial free enterprise in America as we know it would have been impossible. The essence of our American industrial strength lies in our methods of mass production, that is, in the production of precisely similar objects in great number. This idea was put into practical use as early as the year 1800 by Eli Whitney. It is a curious fact that Whitney is popularly remembered for his invention of the cotton gin rather than for his infinitely more important idea of mass production. No doubt this is because the popular mind is much more readily captured by a spectacular new object than by a profound new idea, no matter how vastly more consequential the latter may be. Many years after Whitney demonstrated his army rifle made of "standard" reproducible parts, it became evident that standardized materials were a prerequisite to standardized products. This was first realized in Europe and gave rise there to the first societies for the testing of materials in the 1880's. From these European beginnings there sprang the American Society for Testing Materials.

PROFOUND CHANGES ARE COMING

In the six decades of its existence, the task of our Society has grown hugely in magnitude but has changed little in principle. Our organization, procedures, and objectives remain much as they were in the beginning. Now we face the necessity of making profound changes as a result of deep underlying human developments which, at least ten years ago, began to loom large in our future. That we have only now begun to adjust our Society to the new circumstances is no reason to feel that we have been laggard. The services and functions that ASTM performs for the nation are so basic and so vital that we must do our best to see that they are done with utmost care and competence. In the past, this has always required that we act with studious deliberation. Our technical committees have always seen to it that no important standard was set until all the available technical facts were checked and rechecked by experiment and by experience.

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* Presented at the 64th Annual Meeting of the Society, Atlantic City, N. J., June 25–30, 1961.

[1] Vice-President, Portland Cement Assn., Chicago, Ill.

Here lies, precisely, one of the profound changes in procedures and objectives that must be made in our work. Technological progress in materials now advances at an immensely greater speed than was the case in the first year of this century when our Society's committee and administrative structure was designed. Nor is this faster progress only a matter of accelerated proliferation of new materials and combinations of materials. That problem could conceivably be met by a comparable proliferation of ASTM committees.

But we have now entered the era of molecular and microstructural design in which new materials may originate not only in experiment and experience but also through mathematical calculation of macrostructural properties from known physical constants of various atomic and molecular species. This approach may yet be far off and, indeed, may never be used for natively heterogeneous mass materials such as portland-cement concrete. But these rough materials which do not require the utmost refinement in useful performance are not those which will most demand the future attention of the American Society for Testing Materials.

SCIENCE MUST SUPPLANT EMPIRICISM

During the six decades since its birth, the ASTM has based its work principally upon the great reservoir of practical experience represented in the Society's membership. Only thus could the Society have achieved its ends of providing American industry with necessary materials standards. But time is essential to experience, and the urgent challenges of today and tomorrow no longer allow us to treat time as a plentiful commodity. It in no way diminishes the importance of the practical man of experience to be working in a team with the materials scientist. Even the mathematician, made fantastically productive by the electronic computer, may be a fountainhead of ASTM standards in our next span of six decades.

Recognition was given to these trends in the birth last year of ASTM's new Division of Materials Sciences wherein the fundamental characteristics common to all materials are explored. From the work of this new Division there will surely result an orderly generic scientific approach to useful materials standards. The mounting flood of technical information, which is now the dominant and unprecedented aspect of our lives, may thus be a source of strength rather than of confusion. Great credit for vision and persistence are due the Society's recent past-presidents Woods, LaQue, and Kropf, who, strengthened by the deep convictions and able assistance of Executive Secretary Robert Painter, founded the Division of Materials Sciences.

As a *separate* Division it can provide, within the uniquely broad framework of ASTM, the world's most useful and inspiring continuous forum for the scientists of all countries who are concerned with materials. In this regard, it would be difficult to conceive of any other agency of equal potential. But if this Sciences Division remains *largely separate* from the rest of ASTM it will fail to fulfill its greater purpose of bringing the new knowledge from the research laboratory directly to the major task of improving the standards necessary to our national industrial life. The truly revolutionary aspect of modern science is its pervasive and dominating immediacy to all phases of western civilization. This immediacy of science to living ASTM standards must be consciously and actively insured by effective liaison between our Division of Materials Sciences and our technical committees which are responsible for standards. I charge my successors in the presidency with the task of developing formal organizational means to this end.

The explosive expansion of science and technology is the basic motivating and controlling force in the 20th-century development of American life. All other forces toward change are derivative and secondary. But some of these are so direct that they can be considered separately. Foremost among these is the vast increase in wealth which arises from the broadening mass applications of advancing science and technology. These mass production procedures are possible only if the resulting wealth is widely distributed. This is just a way of saying that practically everyone in America is now so well paid that we can afford to hire no one to labor in the old unproductive traditional ways. The route from raw material to finished product is therefore made as short as possible and all intermediate labor is eliminated or diminished to the minimum. As part of this trend, the materials producer tends to be a manufacturer of semifinished products ready for final assembly. This may or may not lessen the demand for standardization of materials, but it certainly increases the pressure for standards governing intermediate or end products. Thus we find ASTM developing standards not only for plastics but also for zippers, not only for cements and concretes but also for pavements of standard skid resistance! Life promises to become complex indeed for an ASTM which is already the world's most complicated technical society. The industrial diplomacy which our successors must exercise in the committees, the boards, and the offices of ASTM will have to be of at least as high quality as that devoted to our nation's international relationships. Let no cynic express the hope that it may be even higher.

The onrushing tides of science and technology have also made inevitable an augmented partnership between industry and government which will become much wider and more intimate. Man's new mastery of nature, both in infinite space and in the infinitesimal atomic nucleus, gives him technical powers too great to be exercised by any organization less than the nation as a whole. Indeed, it is already evident that the world's entire society of nations is the smallest organization that can govern many of mankind's most important activities in the next sixty years. The more ardent individualists among us will, of course, resent and resist this development. My own father would have been one of these. He was sure that his beloved steel industry could never survive the eight-hour day when, in effect, this was imposed by government. But, fortunately, man's adaptability to change knows no limits.

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THE DEMOCRATIC WAY MUST PREVAIL

Over the past year, as most of you know, I have spoken to many audiences concerning my experiences during the summer of 1960 when, as chairman of an American construction delegation, I had an opportunity to travel widely and with remarkable freedom about the Soviet Union. I have outlined the notable success of the Soviet government in urbanizing and industrializing their huge nation. I have emphasized the central and indispensable part which standardization plays in their fast-mounting industrial strength. From their many central and regional governmental laboratories and academies all standards are issued and all materials progress is directed. The transition of research findings into practical use takes place speedily and efficiently.

Can we match the ultimate Soviet rate of industrial growth? As of 1961 no one can say. Neither their steady growth potential nor ours has been determined, and as of this moment the situations in America and in the Soviet Union provide no basis for economic comparison. We are struggling against a surfeit of technological success which has no historic precedent. We are choking on a uniquely rich diet of overproductivity which we have not found a way to digest. The Soviets, on the other hand, are struggling to emerge from 800 yr of an almost continuously dark and tragic history. Beginning with the twelfth-century conquest of all Russia by the Mongol hordes of Ghengis Khan, the people of the Soviet lands have known little but enforced servitude and medieval bondage. Almost untouched by the enlightenment of the Renaissance or the humanism of the Reformation, the people of Russia think that, through science and technology, they now see their way for the first time to salvation. They have an all-absorbing goal—a decent standard of living—which they are fiercely determined to reach. They think they see their way to it and—I urge you to realize—they are moving fast along that way.

What does this have to do with ASTM? A great deal, I believe. In a talk which I have given to numerous ASTM District Councils I have pointed out that our Society is almost the perfect example of "Democracy in Action."

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I have traced the steps by which democracy originated in urban life, how this urban democracy led to free enterprise and thus on to modern industrialization which, in America, has become synonymous with the mass production of wealth. Finally, mass production is made possible only through standardization. Thus we find America's central society for standards, ASTM, at the very source of our industrial democracy.

The American Society for Testing Materials is a voluntary association of private individuals, private organizations, educational institutions, and governmental agencies, come together to serve this nation and those of other nations who generously work with us. We all combine our efforts as equals in the vineyard, and the fruit of our labor is freely available to any who choose to take it. This is the way of free enterprise and democracy. To those who do their fair share in the work of ASTM I say, "Well done." To those who go beyond the strict call of duty—and there are more than a few of these—I say, "We are thankful for you and your kind, for it is such as you who have made our way of life possible."

To those who do less than their share of the work—or none at all—I say, "Don't sing a song of free enterprise when you are taking a free ride, for this is the funeral music of democracy."

We face a massive demand to prove that our way of progress is best. The people of the Soviet Union know what they want and they believe they know how to get it. With utmost vigor and speed, they are pressing science into the service of their nation. We cannot quarrel with them for this. It is for us to show that we can do as well, or better, in that task. Industrial materials standards spring quickly from the research laboratory into field use by the Soviet system. ASTM must match that level of effectiveness. If we do not, but become, instead, an impediment to technological progress, then we shall deserve extinction. And when that happens, America will be consciously or unconsciously on its way to the Russian system of governmental industrial control.

ASTM must maintain an alertness to progress and a readiness to change second to no other organization in America. I hope we shall do this not so that we may "beat the Russians" in a race for industrial super-productivity. We have already won that race. The best the Russians can now do is to catch up to us as we continue to move on. I believe we should hope that not only they but all other peoples of the world may likewise "catch up with us" and come to know our absence of hunger and poverty. When we can all take our minds off the struggle for a plentiful material existence, we shall then be able to look to the nobler ends which are within man's capacity and which must be his destiny.

From a higher hill one should be able to see more distant horizons. From the eminence of the ASTM presidency which it has been my privilege to occupy this year, I have tried to discern what lies beyond the immediate forest of ASTM problems and challenges. We have been patiently making our way by unhurried winding paths through the familiar terrain. But not far off there loom some formidable cliffs, and beyond these are mounting ridges which rise to spectacularly high and rugged ranges of new knowledge on the horizon. It is evident that we must conquer these if we are to break out of the confines of the Present. It is obvious that to do this some of us must put on the wings of Science and establish advanced bases high up on the Atomic

Cliffs and on the Molecular Mountains. Some must continue to work at extending the pedestrian paths of Experience. But let us never forget that there are other societies and tribes which are eager to reach and occupy the distant summits ahead of us. They will use not only wings but also rockets to span Time, Space, and Matter.

From the president's chair in 1961, I cannot see the routes that ASTM will open up and follow in order to command the scientific and technological heights of 2021. But I see very clearly that the old ways which have served us so well during our first 60 years will be hopelessly inadequate for our next 60. The exponential expansion of our scientific frontiers through research and development will continue unabated. There are a few here assembled who can remember the year 1901, when ASTM was conceived. They know that only since that day we have learned how to release the fundamental forces which move the universe *external* to man. Those here who will eventually remember the year 2001 will know by then that man can release and use his own *internal* capacities to even greater effect upon his own future. We, the present workers in ASTM, are given the privilege of participating in the freeing of man from his own limitations. The future is as bright as we choose to make it.

1961 ANNUAL REPORT OF THE BOARD OF DIRECTORS

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1960–61 was a year of considerable import for the Society. The changes which took place during the year included not only new technical activities, but changes in administration which will have a long-range effect on the Society. These changes give evidence of the dynamic strength of ASTM.

The symposia conducted by the new Division of Materials Sciences at the Annual Meeting in 1960 were outstandingly successful. With this encouragement the Division is moving ahead with plans to add to the store of fundamental knowledge of basic engineering materials, thus fulfilling one of the Society's primary objectives.

New areas of technical activity in which work was started this year include such varied subjects as sensory evaluation of materials and products, slide fasteners, gas chromatography, and vermiculite concrete. In addition, the scopes, titles, and jurisdictions of a number of older technical committees were reviewed so that they may maintain a position of leadership in their fields.

In district activities there was a continued high level of growth—three new districts were established during the year.

In looking ahead with plans for the national meetings of the Society, the Board accepted an invitation to hold its 1968 Annual Meeting in San Francisco. This will be the first Annual Meeting of the Society to be held west of the Mississippi River.

In membership the Society is continuing to show a steady growth—its present membership is 10,505.

A review of the financial position of the Society highlights the soundness of its fiscal operations. Of particular interest during the past year was the considerable improvement in actual operations over the budgeted operations—a gain of slightly more than $64,000.

The past year also saw the retirement of R. J. Painter from active leadership of the Society as its Executive Secretary because of continued ill health. The Board is hopeful of his continued support, when possible, as Consultant to the new Executive Secretary T. A. Marshall, Jr.

But what of the future? In looking ahead, the Board is reviewing the relationship of Staff services to the technical committees and is considering plans for Headquarters expansion. In addition, the first phases of the Long-Range Planning Committee's report to the Board of Directors have now been submitted. We can look ahead to continued growth in services to members, to the American industrial economy, and to the general welfare of the Nation.

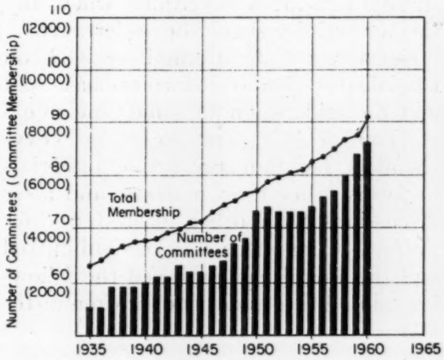
Technical Activities

No attempt is made here to report in detail the technical activities of the Society other than to state that these activities have been at a high level in the production of both standards and technical publications. The standards activities are reviewed in the Report of the Administrative Committee on Standards appended hereto.[1] The record of actions on standards set forth in this report show that the actions taken at the 1960 Annual Meeting resulted in 132 new standards making a total of 2765. During the year the Standards Committee approved 56 additional tentatives. This report also includes mention of the organization of new committees and other extensions of the standardization work. Recorded below are specific actions of the Board with respect to the establishment of new committees and on adjustments of official statements of scope where extensions or clarifications seemed desirable.

Of especial interest are the new areas of coverage, and in such instances the Board must satisfy itself that the new work is justified. With the organization of each new committee, however, the importance of providing adequate coordination assumes ever greater importance, and the matter of coordination is one that is receiving considerable attention and enters into the considerations in establishing or modifying the scopes of activities of committees to avoid duplication of effort. This is especially true in the relation of the general methods of test committees to the work of the product committees.

Revised Committee Scopes, Titles, and Jurisdiction:

Committee B-5.—The Board has approved a rewording of the scope of Committee B-5 on Copper and Copper Alloys, Cast and Wrought, to read as indicated below. There is no change in substance.



Showing Growth in Number of Committees and in Official Committee Membership.

"The formulation and standardization of specifications, the development of engineering information and methods of test, and the stimulation of research relating to copper and copper-base alloy castings and wrought products.

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[1] See p. 87.

"The Committee participates in joint action with other committees in matters of mutual interest.

"Castings here include ingots for remelting, deoxidizers, and hardeners for copper and copper-base alloys and cast products.

"Wrought products include fabricated flat products (plate, bar, sheet, strip, foil and flat wire), rod, wire, shapes, forgings, pipe and tubular products, cartridge case, and bullet jacket cups.

"Products excluded are cathodes, cakes, slabs, billets, wire bars, die castings, and wire specified for use as electrical conductors."

Committee C-2.—Committee C-2 on Magnesium Oxychloride and Magnesium Oxysulfate Cements has no active projects before it. The committee has accordingly been given an inactive status, continuing a small group to be responsible for the review of the present standards and to determine when any further activity should be undertaken.

Committee C-3.—Committee C-3 on Chemical-Resistant Mortars has had under consideration for some time an expansion of its scope so as to cover chemical resistant protective materials in forms other than mortars, and after discussions with other interested committees, Committee C-3 has submitted and the Board has approved the following revised title and scope for Committee C-3:

Title: Committee C-3 on Chemical Resistant Non-Metallic Protective Materials.

Scope: The formulation of methods of testing, specifications, definitions, recommended practices and the stimulation of research relating to chemical resistant nonmetallic materials used primarily for protection against corrosive chemicals. The activities will be coordinated with those of other related committees of ASTM.

Chemical-resistant materials here include mortars, hot-melt compounds, and sheet- and self-supporting liners.

Committee D-9.—In revising its bylaws, Committee D-9 on Electrical Insulating Materials suggested that part (1) of its scope be editorially changed to read as follows, and the Board has given its approval:

"(1) Development of methods of sampling and testing, and formulation of specifications for solid electrical insulating materials, insulating varnishes, and any other insulating materials that are solid in normal use even though they may be applied as liquids. Materials comprise both those designed as electrical insulation and those grades of other dielectric solids designed with controlled dielectric, piezoelectric, or magnetostrictive properties."

Committee D-10.—Because its present title does not reflect the current or future scope of the committee's activities, the title of Committee D-10 on Shipping Containers has been changed by Board action to Committee D-10 on Packaging. In view of the activities of Committee F-2 on Flexible Barrier Materials in the packaging field, the Board has also approved the addition of the following sentence at the conclusion of the present scope of Committee D-10:

"The responsibility for the development of standards for the basic materials used in packaging shall in general rest with the material committees."

Committee D-13.—To include in its statement of scope provision for the preparation of recommended practices respecting textile materials and products, the scope of Committee D-13 on Textile Materials has been revised to read as follows:

"To formulate (1) specifications, (2) methods of test, (3) definitions, and (4) recommended practices respecting textile materials and products, and to promote the knowledge of the properties of textile and related materials."

Committee D-20.—The Board has approved a revised scope for Committee D-20 on Plastics as follows:

"The development of test methods, specifications, recommended practices, nomenclature, definitions, and the stimulation of research relating to plastics; their raw materials, components, and compounding ingredients; and to finished products made from plastics, such as sheets, rods, tubes, pipe, cellular materials, and molded or fabricated articles.

Committee E-1.—The following revised scope for Committee E-1 on Methods of Testing has been approved:

"(*a*) To aid and advise the technical committees of the Society in the preparation of standard methods and development of apparatus of testing, in the correlation of similar methods prepared by different committees, and in instituting cooperation between the technical committees on matters of common interest relating to methods and apparatus of testing, by the establishment of subcommittees of Committee E 1 or otherwise.

"(*b*) To prepare specifications for apparatus for testing materials and methods of testing materials either not included in the scope of other ASTM technical committees or at the request of one or more interested technical committees of the Society, and to initiate, conduct, and report related work upon the determination of properties of materials.

"(*c*) To encourage, secure and promote, with the approval of the Board of Directors, cooperation between technical committees of this Society and the committees of other societies and associations as regards methods and apparatus of testing.

"(*d*) To standardize the nomenclature and definitions used in or relating to testing methods or apparatus in cooperation with Committee E-8 on Nomenclature and Definitions.

"(*e*) To perform such other functions as may advance, improve, standardize, and unify the procedures of materials testing."

Special Committee on Fracture Testing of High-Strength Sheet Materials.—The committee's title has been modified by the substitution of the word "metallic" for the word "sheet" since its work has reached a point where it is felt it should be broadened to include materials other than sheet. The revised title reads as follows: "Special Committee on Fracture Testing of High-Strength Metallic Materials."

It is understood that if the committee prepares any test methods they will be referred either to an existing ASTM technical committee for promulgation, such as Committee E-1 on Methods of Testing, or the special committee could be reconstituted as an ASTM technical committee for this purpose.

Magnet Steels.—Certain developments in the International Electrotechnical Commission (IEC) suggested the desirability of having more complete ASTM specifications for magnet steels. Such specifications are under the jurisdiction of Committee A-6 on Magnetic Properties. They are of general interest to Committee A-1 on Steel and that committee is appointing several representatives to a new Subcommittee on Electrical Steels that is being set up under Committee A-6.

Wrought and Cast Alloys for Structural Use at High Temperatures.—Work on wrought and cast alloys for structural use at high temperatures has been transferred from Committee B-4 on Metallic Materials for Thermostats for Electrical Resistance, Heating and Contacts, to Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys. This includes also the transfer of jurisdiction of Specifications for Chromium-Nickel-Iron Alloy Castings for High-Temperature Service, 25-12 class (B 190 – 50) and Specifications for Nickel-Chromium-Iron Alloy Castings for High-Temperature Service, 35-15 class (B 207 – 50).

Committee D-11 on Rubber and Rubber-Like Materials and Committee D-20 on Plastics cover materials where it is difficult to define precisely the areas of jurisdiction between the two committees. Meetings of the officers are held for the purpose of reaching agreement on assignments and in establishing joint task groups where these can function to advantage. Such a group on Definitions of Terms has an extremely important assignment and serves under the chairmanship of a member of the Staff.

New Activities:

Sensory Evaluation of Materials and Products.—Mention was made in the report a year ago of the authorization for a new Committee E-18 on Sensory Evaluation of Materials and Products. The organization meeting of this committee was held on October 12, 1960.

Slide Fasteners.—Committee D-13 on Textile Materials has had under development in its Subcommittee on Ultimate Consumer Textile Products standards covering definitions and test methods for slide fasteners. This subject had been brought to the Board in view of the question as to whether this type of standard was within the scope of the Society's activities since it involved to some extent engineering design. The Board has considered the submittal to the Society of these standards to be entirely in order, subject, however, to the review of the Administrative Committee on Standards. It is expected that these standards will be submitted to the Society by Committee D-13 in the near future.

Vermiculite Concrete.—The Society has been approached by the Vermiculite Institute with respect to undertaking work on specifications for vermiculite concrete. It developed that the specifications contemplated related largely to design and installation and in view of this would fall outside the general scope of ASTM activities. A sectional committee has been authorized under the American Standards Assn. with the Vermiculite Institute as sponsor. It is understood the Society will be asked to serve as cosponsor.

Gas Chromatography.—The subject of Gas Chromatography has been receiving consideration in several of the Society's committees, including Committee E-15 on Analysis and Testing of Industrial Chemicals. In considering the establishment of a new subcommittee under Committee E-15 it developed that the subject was of such broad significance and interest that it warranted the establishment of a new separate technical committee. This recommendation was brought to the Board, which authorized a new committee on the subject. An organization meeting of Committee E-19 on Gas Chromatography was held on February 6, 1961, with the following scope:

> "To promote the development and application of Gas Chromatography by:
>
> (*a*) Coordinating scientific applications and methods of analysis based on gas chromatography in cooperation with other technical committees of the Society and other organizations.
>
> (*b*) Cooperating with national and international bodies in standardizing (1) presentation of data and (2) nomenclature.
>
> (*c*) Developing general methods and other appropriate standards.
>
> (*d*) Providing a forum for exchange of information.
>
> (*e*) Stimulating research in this field.
>
> (*f*) Collecting and disseminating critical data."

National and Other Meetings

1960 Annual Meeting:

The Sixty-third Meeting of ASTM in Atlantic City, N. J., June 26 to July 1, 1960, again broke all records for attendance with a total registration of 3339. Over 1000 meetings of technical committes were held. The program consisted of 37 technical sessions, the President's Luncheon, the Annual Dinner, and special functions sponsored by the Division of Materials Sciences. These included two symposia, both dealing with fundamentals of materials and a "Materials Science Luncheon" at which W. O. Baker, Vice-President of Bell Telephone Laboratories, spoke on "The National Role of Materials Research and Development." The committee reports presented, as well as a number of the technical papers, are published in the 1960 *Proceedings*. Other papers, particularly those comprising symposia, appear in various issues of the ASTM BULLETIN and as special technical publications (STP's). A review of the meeting appeared in the July, 1960, ASTM BULLETIN.

District Support.—The Philadelphia District again was host for the Annual Meeting in Atlantic City. With the greater proportion of national meetings being held in Atlantic City, the Society is repeatedly indebted to the Philadelphia District for its support. The District Council's efforts were outstanding in providing the very excellent ladies' entertainment. Again, a most welcome break in the week of intensive committee meetings was provided by the Philadelphia District in the form of a cocktail party, entertainment, and dinner on Wednesday evening.

1961 Committee Week:

The Ohio Valley District Council acted as host to Committee Week, and on Wednesday sponsored a cocktail party and dinner. Following the dinner, Mr. Bernard B. Berger, Chief, Water Supply and Pollution Control Research, Robert A. Taft Sanitary Engineering Center, presented a most interesting talk on "How Effectively Can We Control Water Quality?"

Some 350 meetings of committees and subcommittees were held. A symposium on Fire Test Methods, under the auspices of ASTM Committee E-5 on Fire Tests of Materials and Constructions, was held Wednesday afternoon. The symposium consisted of four very interesting papers which, together with discussion, will be published as *ASTM STP No. 301.*

National Technical Meeting—Symposium on Materials and Electron Device Processing:

An innovation that may be establishing a worthwhile precedent is the holding of special national meetings, such as that held in Philadelphia, April 5–7, 1961. The technical program was organized by Committee F-1 on Materials for Electron Tubes and Semiconductor Devices and comprised a three-day Symposium on Materials and Electron Device Processing[2] with the presentation of some 35 papers by authors reporting on research throughout the world. The attendance of over 300 was drawn from all parts of the country, with a good representation from abroad. A dinner sponsored by the Philadelphia District was held on April 6 at which John W. Campbell, Editor, Analog—Science Fact and Fiction, spoke on the subject "Orthodoxy in Modern Science."

Future Meetings:

Annual Meetings of the Society have been scheduled as follows:

June 24–29, 1962—New York, N. Y.
June 23–28, 1963—Atlantic City, N. J.
June 21–26, 1964—Chicago, Ill.
June 13–18, 1965—Purdue University, Lafayette, Ind.
June 26–July 1, 1966—Atlantic City, N. J.
June 25–30, 1967—Boston, Mass.
June 16–21, 1968—San Francisco, Calif.

Committee Week and Spring Meetings are as follows:

February 5–9, 1962—Dallas, Tex.
February 4–8, 1963—Montreal, Quebec, Canada.
February 3–7, 1964—Philadelphia, Pa.
February 8–12, 1965—Cleveland, Ohio.
January 30–February 4, 1966—Washington, D. C.

Pacific Area National Meetings are scheduled as follows:

September 30–October 5, 1962—Los Angeles, Calif.
October 31–November 5, 1965—Seattle, Wash.

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[2] To be published as *ASTM STP No. 300.*

Publications

In the report a year ago mention was made of the decision of the Board that the ASTM BULLETIN beginning in 1960 be issued monthly instead of eight times a year as at present. During the year the Board also authorized that in making this change the format and title be changed as well. The new title is *Materials Research & Standards*. Placing it on a monthly basis will provide a more frequent means of bringing news of the Society to the attention of its members. The other changes will result in placing more emphasis on the publication of technical material.

One other matter, also referred to in the report a year ago, was the decision to issue the 1961 Book of Standards in eleven rather than ten parts as in the previous edition. However, as the report of the Publications Committee indicates, studies are now going forward looking toward a complete realignment of the Book of Standards with a possibility of issuing it in a far greater number of parts, corresponding to the special compilations that are now made available. Such a move, while involving quite a number of adjustments that would need consideration, could result in eliminating some duplication of printing and thus lightening and evening out the editorial and printing load.

The increasing number of meetings that are being sponsored naturally results in increased publications, either by way of articles in *Materials Research & Standards* or in separate publications.

District and Related Activities

Through its districts, the members and committee members of ASTM are provided with the opportunity to participate at the local level in the affairs of the Society. During this year the districting of the Society to cover all of the continental United States was completed with the formation of three new districts. These three districts bring the total number now to 20. The new districts are the Northwest District, composed of British Columbia, and the States of Washington, Oregon, Idaho and Montana; the Central Plains District, composed of Kansas, Nebraska, Iowa, and western Missouri; and the Northern Plains District, composed of North Dakota, South Dakota, Minnesota, and western Wisconsin. Consideration is also being given to the formation of a North Central Texas District.

One of the important activities of the districts has been the presentation of student membership awards to outstanding students in the engineering and technical colleges. Many of these are presented at district meetings, during which these students who have demonstrated ability and special interest in materials are brought into contact with the established members of the Society. The Washington District sponsored a novel program having these students particularly in mind. This program consisted of a special luncheon at which speakers described ASTM and its activities and the National Bureau of Standards. This was followed by a tour of the facilities of the NBS.

President Bates was the principal speaker at 19 district meetings; in addition he spoke before numerous civic and business clubs in conjunction with these appearances. Vice-Presidents Clair and Seniff and Past-President Woods each addressed one district meeting.

The variety of topics offered at district meetings this year is worthy of note. Several full-day conferences were sponsored, including two held by the New England District on "Current Materials Problems" and "Automation." The Rocky Mountain District sponsored an all-day session devoted to Concrete jointly with the American Concrete Inst. In addition, the Philadelphia District cooperated in sponsoring the first National Technical Meeting of the Society in cooperation with Committee F-1 on Materials for Electron Tubes and Semiconductor Devices.

A list of the district meetings during the year follows. Most of these meetings were held in cooperation with other technical groups, and news accounts were reported during the year in *Materials Research & Standards.*

District Meetings:

District	*Date*	*Location*
Southern California District (jointly with AFM)	September 22, 1960	Los Angeles, Calif.
Central New York District (jointly with AFM)	October 10, 1960	Corning, N. Y.
New England District	October 27, 1960	Boston, Mass.
Mississippi Valley District (jointly with Engineers Club of St. Louis)	October 27, 1960	St. Louis, Mo.
New York District (jointly with the Chemists Club)	October 27, 1960	New York City
Rocky Mountain District (jointly with ACI)	October 31 to November 2, 1960	Tucson, Ariz.
Philadelphia District	November 9, 1960	University of Delaware, Newark, Del.
Washington District (jointly with Committee E-6)	November 14, 1960	Washington, D.C.
Detroit District	November 17, 1960	Detroit, Mich.
Philadelphia District (jointly with ASQC)	January 19, 1961	Germantown, Pa.
Southern California District (jointly with ACI)	January 19, 1961	Los Angeles, Calif.
Pittsburgh District (jointly with SNT)	January 19, 1961	Pittsburgh, Pa.
Northern California District (jointly with ACI)	January 23, 1961	San Francisco, Calif.
Northwest District	January 25, 1961	Seattle, Wash.
Ohio Valley District (in conjunction with Committee Week)	February 1, 1961	Cincinnati, Ohio
Washington District	February 3, 1961	Baltimore, Md.
Northern California District	February 23, 1961	San Francisco, Calif.
Southwest District (jointly with ASCE)	February 24, 1961	Austin, Tex.
Central New York District (jointly with ASM)	March 6, 1961	Rome, N. Y.
Southeast District (jointly with ASM)	March 7, 1961	Birmingham, Ala.
Southwest District (jointly with AIA)	March 8, 1961	New Orleans, La.
Southwest District (jointly with ASCE)	March 9, 1961	Houston, Tex.
Southwest District (jointly with ACS)	March 11, 1961	Bartlesville, Okla.
Cleveland District (jointly with ASCE and Cleveland Engineering Society)	March 30, 1961	Cleveland, Ohio
Philadelphia District (jointly with Committee F-1)	Arpil 6, 1961	Philadelphia, Pa.
Washington District (jointly with Raleigh Engineers Club)	April 10, 1961	Raleigh, N. C.
Southwest District (jointly with TSPE, ASCE, and CSI)	April 10, 1961	Dallas, Tex.
Chicago District (jointly with ASTM Committee D-10)	April 11, 1961	Chicago, Ill.
New York District (jointly with ASCE and CIB)	April 19, 1961	New York City
Western New York - Ontario District	April 20, 1961	Niagara Falls, N. Y.

District Meetings (*Continued:*)

District	*Date*	*Location*
Detroit District (jointly with ASCE and ACI)	April 25, 1961	Detroit, Mich.
Pittsburgh District	April 26, 1961	Pittsburgh, Pa.
Washington District	April 28, 1961	Washington, D. C.
New England District (jointly with Maine Association of Engineers, MSPE, ASCE, and AIEE)	May 6, 1961	University of Maine, Orono, Maine
Northwest District (jointly sponsored with several technical societies)	June 13, 1961	Richland, Wash.

Membership

New members continue to join the Society at more than a thousand per year. In the period May 1, 1960, to May 1, 1961, more applications were accepted than in any previous year—1130—as compared with 1100 in 1959, the previous high year. However, net growth is always substantially less because of losses from death, resignation, and nonpayment of dues. The losses reported this year are higher than for previous years due to a decision to adhere to the By-laws by dropping members whose dues had not been paid in the one-year period allowed. During the depression, members with dues in arrears were given an additional seven months to pay before being dropped. This year, delinquent members were dropped January 1, 1961, one year from the date their dues were due, as the By-laws provide. As a result the records show a greater loss than usual. The net gain in new members was 242 in all classes except student, as compared with 477 a year ago. If adjusted to take into account only one year's loss, the net gain would have been 478.

While the membership growth moves steadily upward, the Membership Committee and the Board of Directors are concerned that the relative growth of the Society in members is not keeping abreast of the growth of scientific and engineering activities in our economy, or of its own growth in technical committee activity and publications. The Board has authorized additional personnel to enable the staff to give more attention to the promotion of membership in all classes, but especially the company grade; and to promote the knowledge and use of the Society's standards and publications, which in turn are excellent stimuli to growth.

On May 1, 1961, the membership of the Society totaled 10,505, compared with a total of 10,263 one year ago. Gains and losses in various classes of membership are shown in the following table:

Class of Membership	Membership		Losses				Additions		Total		
	May 1, 1960	May 1, 1961	Resignations	Dropped	Death	Transfer	Transfer	Election	Loss	Gain	Increase
Honorary	27	27	...	...	...	...	...	...	...	...	...
Perpetuity and Life	13	14	...	...	...	...	...	1	...	1	1
Sustaining	407	433	6	2	...	6	38	2	14	40	26
Company, Firm, etc.	2 094	2 040	71	44	...	51	8	104	166	112	54[b]
Individual, etc.	7 364	7 599	358	286	52	3	63	871	699	934	235
Associate	358	392	20	48	1	50	1	152	119	153	34
Total	10 263	10 505	455	380[a]	53	110	110	1130	998	1240	242
Student	1 637	1 757	501	372	...	83	...	1076	956	1076	120

[a] Represents two years due to change in date for dropping of delinquent members.

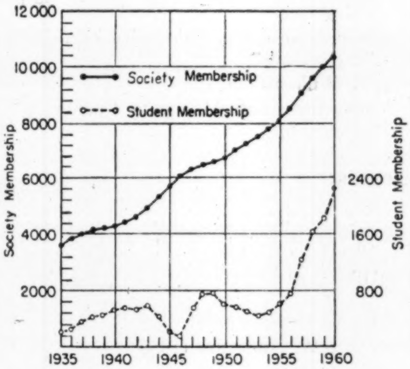
[b] Net loss.

It is interesting to compare our present membership of 10,505 with the figures for ten and twenty years ago; in 1951 our total membership was 7001, and in 1941 it was 4470.

Associate and Student Memberships:

The class of Associate Membership, which continues to 30 years of age and replaces the former junior membership with its 27-year age limit, continues to grow as noted in the table.

Because of the influence of student membership prize awards, which recognize outstanding students in courses related to the Society's work, the very encouraging growth in this class has continued. The Society itself and many of the districts, in addition to individuals and companies, are sponsoring student membership awards.



Showing Growth in Membership

Sustaining Members:

Forty new sustaining members, largely transfers from company membership grade as a result of a special invitation from our president, more than offset the 14 losses in this class. The new sustaining members are:

Amoco Chemicals Corp.
Atlantic Steel Co.
The Beryllium Corp.
Black & Decker Manufacturing Co.
Calaveras Cement Co.
Canada Iron Foundries, Ltd.
Canadian Industries, Ltd.
Canadian Pacific Railway Co.
Certain-teed Products Corp.
The Chemstrand Corp.
Dominion Foundries and Steel, Ltd.
Electrical Testing Laboratories, Inc.
Enjay Chemical Co., Inc.
The Ensign-Bickford Co.
Goodrich-Gulf Chemicals, Inc.
Goodyear Atomic Corp., Library
W. R. Grace and Co., Research Div.
Grumman Aircraft Engineering Corp.
Ideal Cement Co.
The Kaman Aircraft Corp.
Keasbey & Mattison Co.
Mallinkrodt Chemical Works
Mobay Chemical Co.
Mueller Co.
Naugatuck Chemical Div., U. S. Rubber Co.
Otis Elevator Co.
Roanoke Electric Steel Corp.
H. H. Robertson Co.
Sandusky Foundry and Machine Co.
Spaulding Fibre Co., Inc.
Stackpole Carbon Co.
J. P. Stevens and Co., Inc.
Stockham Valves & Fittings
Texas Instruments, Inc.
The Trubek Laboratories, Inc.
United Engineering and Foundry Co.
United States Testing Co.
West Point Manufacturing Co.
Wheeling Steel Corp.
C. K. Williams and Co.

60-Year Members:

The list of organizations which have held membership in the Society for 60 years or more is growing and is reported for the first time in this annual report:

American Foundrymen's Society (1898)
American Steel and Wire Div., United States Steel Corp. (1900)
Bethelehem Steel Co., Inc. (1898)
Booth, Garrett & Blair Engineers and Chemists (1896)
The Colorado Fuel and Iron Corp. (1900)
Franklin Institute (1898)
Robert W. Hunt Co., Engineers (1899)
The Lowe Brothers Co. (1899)
National Tube Div., United States Steel Corp. (1900)
John A. Roebling's Sons Div., The Colorado Fuel and Iron Corp. (1900)
United States Steel Corp. (1898)

50-Year Members:

There are three individuals and fourteen organizations as noted below who this year have been members of the Society continuously for 50 years:

S. H. Graf
S. H. Ingberg
Monroe L. Patzig

Alco Products, Inc.
Baldwin-Lima-Hamilton Corp.
Bridgeport Brass Co.
Carnegie Institute of Technology
General Motors Corp.
Hercules Cement Co., Division of American Cement Corp.
Ledoux and Co.
Northern Electric Co., Ltd.
Pratt & Lambert, Inc.
The Steel Co. of Canada, Ltd.
U. S. Department of the Navy, Bureau of Naval Weapons
U. S. Department of the Navy, Bureau of Ships
United States Gypsum Co.
U. S. Naval Engineering Experiment Station

40-Year Members:

There are 51 members who this year have completed 40 years of membership:

Arthur F. Brown
J. F. Calef
Carl M. Duff
F. Malcolm Farmer
Raymond E. Hess
J. J. Laudig
Albert W. Luhrs
Vincent T. Malcolm
John W. McBurney
Alvord P. Meng
Andre Millot
Mortimer F. Sayre
W. A. Selvig
Sam Tour
J. C. Witt

American Ceramic Society, Inc.
Atlas Powder Co.
Bareco Wax Co., A Division of Petrolite Corp.
Boston Edison Co.
Bridgeport Public Library and Reading Room
The British Electrical and Allied Industries Research Assn.
The Budd Co.
California Institute of Technology
Canada Department of Mines and Technical Surveys, Mines Branch
Esso Research and Engineering Co.
B. F. Goodrich Footwear and Flooring Co., Division of The B. F. Goodrich Co.
Hercules Powder Co., Research Center
Houdaille Construction Materials, Inc.
Houston Public Library
Kansas City Public Library
Kansas State University, Road Materials Laboratory
Koppers Co., Inc., Tar Products Div.
Manitoba, University of, Engineering Library
Marquette University, College of Engineering
Missouri Pacific Railroad Co.
Missouri, University of
New York State Library
Northwestern States Portland Cement Co.
H. C. Nutting Co.
The Ohio Steel Foundry Co.
Philadelphia, The Free Library of, Serials Section
Pittsburgh Steel Co.
Precision Thermometer and Instrument Co.
Sheffield Div., Armco Steel Corp.
Solvay Process Div., Allied Chemical Corp.
South Dakota School of Mines and Technology
Arthur H. Thomas Co.
Washington, State of, Department of Highways
Western Australia, University of, School of Engineering
J. H. Williams and Co.
Youngstown and Mahoning County, Public Library of

Deaths:

The Society has lost the following members through death (figures in parentheses are dates of membership):

Alfonso Abbate (1959)
Robert W. Anderson (1956)
Max Bachrach (1958)
Jayson C. Balsbaugh (1958)
Alfred C. Barbour (1944)
Carl Bussow (1935)
Harold F. Clemmer (1915)
Clyde C. Connor (1931)
Charles V. Cooper (1960)
Clyde A. Crowley (1941)
Sigfrid Dahlberg (1957)
Herbert A. Davis (1920)
Ward A. Detwiler, II (1959)
John R. Dwyer (1920)
Marion Eppley (1936)
Charles T. Evans, Jr. (1945)
H. J. Eyman (1948)
E. F. Fernes (1957)
Dozier Finley (1911)
Perry J. Freeman (1923)
Dudley K. French (1913)
Marc S. Goldsmith (1955)
A. N. Goodbrand (1949)
John J. Gould (1950)
Albert Haertlein (1921)

C. E. Harness (1959)
W. Hertner (1959)
Harry Hey (1949)
Willard Wellington Hodge (1947)
Roger L. Johnson (1954)
Earl F. Kelley (1926)
Harold R. King (1943)
A. L. Kuehn (1914)
Serge Leoni (1957)
John M. Lessells (1924)
Frances A. McAdam (1940)
Edgar K. Mull (1941)
Leopold J. Nedorostek (1958)
Charles E. Paul (1908)
E. S. Petruniak (1959)
W. P. Putnam (1911)
Sidney Rolle (1941)
John E. Rutzler (1952)
George Sachs (1954)
Lyle R. Sheppard (1960)
John C. Smack (1949)
Ralph Smillie (1929)
Earle C. Smith (1926)
Edwin K. Smith (1924)
F. W. Smither (1912)
Rudolph M. Snyder (1940)
D. B. Steinman (1927)
D. W. Sudell (1954)
Ernest E. Thum (1924)
James F. Tompkins (1960)
Henri P. C. Vanderapelle (1954)
Vito A. Villani (1960)
Walter C. Voss (1932)
Anthony D. Wagner (1944)
John H. Wickersham (1932)
T. F. Wieczorek (1958)
J. Alexander Woodward (1929)

In addition to the above, the following representatives of company members of the Society passed away:

Campion, E. W., The Bonney-Floyd Co.
Theodore Irving Coe, American Institute of Architects
Robert J. Peters, Warner Gear Div., Borg-Warner Corp.
W. W. Roff, Whittaker, Clark & Daniels, Inc.
A. G. Sturrock, Wyckoff Steel Co.
H. C. Swett, Bethlehem Steel Co., Inc., Pacific Coast Div.

Honors and Awards

The Board's Committee on Lectures and Awards has been active in reviewing present Society and Committee Awards and, on its recommendation, the Board has adopted a Policy on New Awards which appears for the first time in the 1960 Year Book. As mentioned in last year's report, a procedure for recognizing outstanding achievements in technical committees has been approved and will be followed as soon as appropriate. The Society's Manual on Special Lectures, Honors, and Awards is in process of revision and these new policies and procedures will be incorporated in the new edition.

Descriptions of the various existing awards as well as the personnel of the lecture and award committees are given in the ASTM Year Book.

Recipients of honors and awards to be given at the 1961 Annual Meeting are indicated here:

Honorary Membership:

Edward J. Albert
Simon Collier
Theo. P. Dresser, Jr.
Stanton Walker
William A. Zinzow

Award of Merit:

John J. Allen
Frank E. Clarke
Thomas E. Eagan
Davis F. Gould
Russell B. Gunia
Francis M. Howell
Richard S. Hunter
James H. Lansing
William Lerch
Robert R. Litehiser
Percy L. Rogers
Anthony M. Schwartz
Arthur G. Scroggie
Hubert R. Snoke
John W. Whittemore
Lyman W. Wood
John S. Worth

Recognition of Technical Papers:

Charles B. Dudley Medal (research).—G. B. Espey, M. H. Jones, and W. F. Brown, Jr. for their paper "The Sharp-Edge-Notch Tensile Strength of Several High-Strength Steel Sheet Alloys."

Richard L. Templin Award (testing).—

E. A. Neppiras for his paper "Techniques and Equipment for Fatigue Testing at Very High Frequencies."

Sam Tour Award (corrosion).—A. K. Graham and H. L. Pinkerton for their paper "Standardizing the Preparation of Electrodeposits on Test Panels for Corrosion Testing."

Sanford E. Thompson Award (concrete, sponsored by Committee C-9).—Leonard Pepper and Bryant Mather for their paper "Effectiveness of Mineral Admixtures in Preventing Excessive Expansion of Concrete Due to Alkali-Aggregate Reaction."

C. A. Hogentogler Award (soils, sponsored by Committee D-18).—C. B. Crawford for his paper "The Influence of Rate of Strain on Effective Stresses in Sensitive Clay."

Recognition of Outstanding Contributions:

Max Hecht Award (industrial water, sponsored by Committee D-19).—Claude K. Rice, Venezuela Gulf Refining Co., Venezuela, S. A.

Harold deWitt Smith Memorial Award (textiles).—Lyman E. Fourt, Assistant Director of Research, Harris Research Laboratories, Inc., Washington, D. C.

ASTM Lectures:

The *Edgar Marburg Lecture* on "The Nucleation and Growth of Ice Crystals" will be presented on Monday afternoon, June 26, at Haddon Hall, Atlantic City, N. J., by Dr. Bruce Chalmers, Gordon McKay Professor of Metallurgy, Harvard University.

The *H. W. Gillett Memorial Lecture* on "Specifications?!." will be presented on Tuesday afternoon, June 27, at Haddon Hall, Atlantic City, N. J., by Dr. A. B. Kinzel, Vice-President-Research, Union Carbide Corp.

Fellowships and Grants-in-Aid

The second ASTM free grant doctoral fellowship was awarded to the Massahusetts Institute of Technology for the 1961–62 academic year. This award, which provides for $1500 for the University and $5000 for the student during the final year of full-time graduate work on his doctor's degree, was given by MIT to Richard H. Krock.

Five $1000 grants-in-aid were made to the University of Illinois, The Pennsylvania State University, Queen's University (Kingston, Ontario), Rutgers University, and Union College. Descriptions of the projects for which the grants-in-aid were made and the names of the recipients were published in the March issue of *Materials Research & Science.*

Finances

Report for 1960:

Full details of the fiscal operation of the Society for 1960 are included in the report of the auditors appearing in Appendix I.[3]

Operating Receipts:

The figure of $1,343,675 budgeted for 1960 was exceeded by $69,200. The total for the year was $1,412,898. Receipts for dues and entrance fees were $385,058 or 27.3 per cent; the sale of publications amounted to $873,102 or 61.8 per cent; miscellaneous sources, namely, advertising, income from investments, exhibits and registration fees, totaled $154,738 or 10.9 per cent. The income from publications for 1960 was less than for 1959 because this is the second year for the sale of the 1958 Book of Standards.

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[3] See p. 73.

Operating Expenses:

Total expenses were $1,420,115, which included $214,500 placed in reserve to complete payment for the 1960 Supplements to the Book of Standards, the 1960 Proceedings (the final bills for which will not be available until 1961), and $100,000 added to the reserve for the 1961 Book of Standards.

Operating Gains:

Gross operating income over expense was $207,282, less funds placed in reserve of $214,500. This shows a net loss for the year of $7218. The estimated loss budgeted at the beginning of the year was $68,325.

Balance Sheet:

On November 30, 1960 the total assets in the General Funds were $1,128,623[4] compared with the $1,102,822 a year ago. The balance sheet takes no account of the inventory of publications in stock. This is consistent with many years' practice. An inventory of the principal technical publications is made regularly for the guidance of the staff in administering the publications operation.

Building Fund:

Assets in the Building Fund at the end of the fiscal year were $450,915. The total loan advance to the Building Fund now stands at $120,325. We are continuing to amortize at the rate of $17,000 per year as a part of headquarters occupancy costs and this item appears as such in the annual operating budget.

Investments:

The operation of the Investment Fund has continued with no changes. The firm of Loomis, Sayles & Co. is still retained as investment counsel. The income from investments is paid semiannually to the General, Building, Research, and other funds in accordance with their pro rata equity. As of November 30, 1960, the book value or cost of the securities in the fund totaled $945,749. Market value was $1,093,440. Total income was $40,973.

1961 Budget:

The accompanying summary of the 1961 budget shows a modest net favorable balance of $1325. If the receipts continue in the favorable percentage as shown in the first three months, there should be a much larger net gain at the end of the fiscal year.

BUDGET SUMMARY—1961.

		Per Cent
Estimated Receipts:		
Dues and Entrance Fees	$393 675	26.4
Publications	943 500	63.3
Miscellaneous	153 300	10.3
TOTAL CURRENT RECEIPTS	1 490 475	100.0
Estimated Expenses:		
Publications	581 500	39.0[a]
Salaries	543 700	36.5
General Office Expense	151 500	10.2
Technical Committee Expense	13 000	0.9
Meetings	26 750	1.8
Headquarters Occupancy Expenses	61 900	4.2
Retirement, Insurance Expenses	72 300	4.8
Miscellaneous	38 500	2.6
TOTAL OPERATING EXPENSES	1 489 150	100.0
ESTIMATED GAIN	1 325	
TOTAL	$1 490 475	

[a] This figure does not include the usual carryover into the following year to provide for the completion of the 1961 Book of Standards which is estimated to be on the order of $200,000. In previous budgets this has been included in the Book of Standards year.

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[4] Attention should be called to the fact that whereas the Balance Sheets for both General Funds and Building Fund as shown in the Auditors' Report are correct in themselves, adding the two together in the consolidated statement brings about a duplication of assets with respect to the Cherry Street and Arch Street properties, inasmuch as the money for purchase of the properties was loaned (not given) to the Building Fund and hence still appears as an asset in General Funds in the form of a note, whereas the property purchased with that money also appears as an asset in the Building Fund.

Administrative Matters

The Board recognized the difficulties in Society administration resulting from the long-continued illness of the Executive Secretary, R. J. Painter. At the Annual Meeting in 1960, R. E. Hess was designated Acting Executive Secretary and a special committee of the Board undertook the task of finding a successor to Mr. Painter.

At the meeting of the Board of Directors in September, 1960, Thomas A. Marshall, Jr., formerly Senior Assistant Secretary of The American Society of Mechanical Engineers, was elected to the position of Executive Secretary of ASTM, effective October 15, 1960. Mr. Painter was retained as Consultant to the Executive Secretary and since that time has devoted his major efforts to working with the Long-Range Planning Committee under the chairmanship of Past-President N. L. Mochel. Mr. Hess continues as Associate Executive Secretary, as Technical Secretary, and Editor-in-Chief, positions he has most ably filled. F. F. Van Atta, formerly Assistant Secretary, was appointed Treasurer with responsibility for the business activities of the Society. A. Q. Mowbray has been designated Editor of *Materials Research & Standards*.

Headquarters:

The matter of providing an adequate headquarters to meet the expanding needs of the Society has been under consideration by the Board for the past two years. A study of the Staff needs in the light of projected activities through the year 1980 has been prepared and it is expected that an early decision will be reached by the Board with respect to selection of site and expansion of headquarters facilities.

Amendments to By-laws:

During the past year the Board has given considerable study to the matter of membership services and the dues structure of the Society. As a result, amendments to the By-laws which modify the services to which a newly elected member is entitled, as well as amendments providing for the conferral of life membership will be submitted to the membership at the Business Meeting to be held in Atlantic City during the 1961 Annual Meeting.

Regulations Governing the Board of Directors:

The Regulations Governing the Board of Directors, including the necessary financial resolutions for the administration of Society affairs, were reviewed by the Board after the appointment of the new Executive Secretary. Minor modifications were made in the Regulations and new resolutions adopted to provide for dual signatures to safeguard the Society's funds. In addition, the custody of the Society's securities was transferred to the Girard Trust Corn Exchange Bank. This latter action should result in a reduction in Staff operating detail at no increased cost to the Society.

Group Insurance for Members:

The charter enrollment period for the new Group Insurance Program for ASTM Members closed on December 1, 1960. As of that date more than 250 applications had been received. The initial program included a loss-of-time plan and a major hospital plan. Provisions were also included for senior members to obtain protection. The Board has approved the adding of a high limit accidental death and dismemberment insurance plan to the existing program. This will provide the members with an opportunity to obtain such protection at group rates which are considerably less than the cost of providing such coverage on an individual basis. The contract for this additional plan has been signed, and announcements were mailed to the members of the Society shortly before the 1961 Annual Meeting.

Name of Society:

The Board reviewed the status of the proposals for changing the name of the Society which have been under study for the past two years. The Society's present name, American Society for Testing Materials, was chosen when the Society was chartered in 1902. The name chosen then reflected the Society's beginning as the American Section of the International Association for Testing Materials. Discussions were held by the President and the Executive Secretary with district councils throughout the country during their visits in the fall of 1960 and the early months of 1961. These discussions served to reemphasize the importance of ASTM as a *materials* society. The membership in these districts were almost unanimous in urging that the initials ASTM and the Society's emblem be retained intact. A recommendation of the Board to change the Society's name, with an appropriate change in the Charter, to "American Society for Testing *and* Materials" will be presented to the membership at the Business Meeting in Atlantic City on June 27, 1961.

Division of Materials Sciences

Organization:

The Division continues to perfect its organization in line with its objectives as set forth in its scope:

> The promotion of knowledge of the fundamental nature of materials. To this end, the division shall encourage and provide for the exchange and dissemination of knowledge through articles, papers, symposia, conferences, and by any other means that may be considered appropriate and effective.
>
> The division will be concerned broadly with the nature and origin of basic properties of materials, that is, the relation of these properties to their structure, and will give special attention to matters common to more than one material or class of materials. Generally, the division will be concerned with specific materials only to the extent that their properties illustrate fundamental principles.

The above scope and a general plan for organization of the the Division were accepted in principle by the Division Council—the Division's governing body—at its meeting during the Society's 1960 Annual Meeting. The by-laws provide for a Division Executive Committee of from seven to twelve members with staff secretary and a Division Council of up to 100 members representing the technical and administrative committees. A number of members-at-large are also included on the council.

The Division Council elected the following officers and members of the Division Executive Committee:

K. B. Woods, Chairman
Richard T. Kropf, Vice-Chairman
A. Allan Bates
M. N. Clair
W. L. Fink
F. L. LaQue
B. J. Lazan
John Sullivan
A. C. Webber
I. V. Williams

Frank Y. Speight was appointed staff secretary on recommendation of the Executive Secretary of the Society.

According to the by-laws, the officers may serve for two successive terms of two years each, with elections held in even-numbered years.

Proposed revisions of the by-laws also enlarge the Division Council by removing the limitation of number of members but with the stipulation that the number of members-at-large may not exceed the number of authorized committee representatives.

The by-laws also provide for a membership roster for the Division so as to allow those members of the Society who are interested in materials sciences to indicate their desire to participate in the Division's activities.

The results of the ballot on the by-laws will be reported to the Division Council at its meeting on June 27, 1961, at the Annual Meeting and further modifications made to resolve any negative votes.

Technical Program:

At the Sixty-third Annual Meeting the Division sponsored two symposia—on Recent Progress in Materials Sciences, and on Nature and Origin of Strength of Materials. Papers from these two symposia have been published under the title "Properties of Crystalline Solids" (*ASTM STP 283*). The two symposia were enthusiastically received by a capacity audience. At the Sixty-fourth Annual Meeting, the Division is sponsoring a two-session symposium on Major Effects of Minor Constituents on Properties of Materials.[5] The five papers will deal with metals, ceramics, polymers, organic liquids and plasmas. The emphasis will be on review and education; broad participation by technical people working in diverse fields is anticipated.

The Division Executive Committee is planning for the future and will sponsor symposia or other events at the three national meetings in 1962—Committee Week in Dallas, the Annual Meeting in New York, and the Pacific Area Meeting in Los Angeles.

Cooperative Activities

American Standards Association:

In addition to the matters reported in the Annual Report of the Administrative Committee on Standards, a change in the Constitution of the American Standards Assn. was submitted to the Member Bodies. This change enlarged the Board of Directors of ASA and revised the system for the nomination of the additional directors. ASTM voted to approve the changed constitution of ASA.

Government Contacts:

A delegation from ASTM headed by the President and including Past-President LaQue and the Executive Secretary met with representatives of the National Bureau of Standards and the International Cooperative Administration in the offices of the Assistant Secretary of Commerce. This meeting was arranged as a result of correspondence between Mr. LaQue and Secretary of Commerce Luther H. Hodges, and was devoted to the desirability of having selected ASTM standards made available in Spanish and Portuguese for use in Latin-American countries. The current administration appeared receptive and is preparing a specific plan of action to implement such a program.

A new ASTM-ASA Advisory Committee to the National Bureau of Standards on Engineering and Related Standards has now been organized, replacing two former advisory committees to the Bureau. The ASTM representatives include Messrs. A. A. Bates, F. L. LaQue, and N. L. Mochel, with M. N. Clair and the Executive Secretary as alternates. The ASA representatives are J. R. Townsend, G. F. Hussey, Jr., and J. W. McNair, with Cyril Ainsworth as alternate. W. A. Wildhack, General Secretary to the Director of the Bureau, is chairman of the committee.

The present ASTM Committee on Government Contacts under the chairmanship of N. L. Mochel continues. This committee has the responsibility for the supervision of all Government contacts.

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[5] To be published as *ASTM STP No. 304.*

Engineers Joint Council:

In January, 1961, the Society became an affiliate of Engineers Joint Council and commenced active participation on a number of EJC committees of importance to ASTM. These include cooperation in technical planning, international relations, and national water policy.

Engineers Joint Council is the federation of major engineering societies which acts as an advisory and coordinating agency to seek out and study matters of mutual interest to its member bodies and recommend parallel action by them, and to foster cooperation among the various branches of engineering for the general welfare. In addition, EJC develops recommendations regarding public policies in those areas where application of engineering will make a substantial contribution. The Council currently has eleven constituent societies, two affiliates, and a number of associates. The Board believes that by becoming affiliated with EJC, ASTM will gain in recognition and stature as a scientific and educational society.

National Research Council:

The Society was represented at the annual meeting of the Division of Engineering and Industrial Research of the National Research Council by W. H. Lutz and Past-President K. B. Woods. At this meeting the Division discussed the impact of patents on research and development, new area planning, and problems created by the rapid growth of science. Under this latter category the Division considered the problems of scientific meetings and the presentation and publication of scientific papers.

International Critical Tables:

Critical tables—tables of carefully evaluated values of properties of materials and physical and chemical constants—were published in the 1920's by McGraw-Hill under National Research Council auspices, with international cooperation as International Critical Tables. These tables have not been brought up to date, but the continuing responsibility for critical tables still resides with the National Research Council and is effected through the Office of Critical Tables. A. V. Astin is chairman of the small executive committee of the OCT and the director is Guy Waddington.

The Society is represented on the OCT advisory committee by M. V. Otis of Tennessee Eastman Co.

Present efforts of the OCT are toward the assembly of a comprehensive index to critical data wherever it may be published. It has already published a Directory of Numerical Data Projects which includes several ASTM projects—on absorption spectral data, and X-ray powder diffraction data. Until recently the OCT had established no position on engineering data on properties of materials. Now, however, it has been definitely stated that the OCT will be concerned with engineering data such as that published under the auspices of the Joint ASTM-ASME Committee on Effect of Temperature on the Properties of Metals. This stated change in policy of OCT places its objectives closer to those of ASTM and raises the question of communication and coordination of critical tables projects within ASTM.

The establishment of an ASTM committee on Critical Tables is under consideration to focus the Society's interest in this subject and to exchange information between the various numerical data projects.

International Contacts:

Pan American Standards Committee.—A round-table discussion on standards in Latin America was held in Buenos Aires in connection with a meeting of Union Panamericana de Asociaciones de Ingenieros (UPADI) in the fall of 1960. Out of this discussion arose renewed interest in the development of the Pan American Standards Committee to fulfill its responsibilities in promoting standards in the South American countries and assisting various countries in establishing their own standards. An organization meeting to adopt a constitution and to propose a program for PASC for the next three years and a plan for financing this operation was held in Montevideo April 24–27, 1961, and attended by 33 delegates representing Argentina, Brazil, Chile, Colombia, Peru, the United States, Uruguay, and Venezuela.

The U. S. delegation was headed by J. R. Townsend, President of the American Standards Assn. and Past-President of ASTM, and included Vice Admiral G. F. Hussey, Jr., U.S.N. retired, Managing Director and Secretary of ASA, T. A. Marshall, Jr., Executive Secretary of ASTM, and Messrs. H. A. Hunnicutt, R. L. Kessler, B. J. Smith, and A. T. McPherson.

Dr. A. Sinay Neves of General Electric, South America, and a Past-President of the Brazilian Standards Assn. (ABNT) was elected president of the reconstituted PASC, and Dr. Paulo Sa, Director of ABNT, was made its permanent Honorary President. Senora Ing. Beatriz Ghirelli de Ciaburri, Executive Director of the Argentine National Standards Organization (IRAM), will serve as provisional Executive Secretary. The headquarters will remain temporarily in Rio de Janeiro.

Argentine Institute for Standardization of Materials.—In response to an invitation for the Society to participate in the 25th anniversary of the Argentine Institute for Standardization of Materials (IRAM) a scroll was transmitted by the Society and R. J. Painter prepared a paper on "Research and Standardization for Materials in the USA" which was included in the program and subsequently published in the regular journal of IRAM.

Other Activities.—Highlighting a number of activities in other areas are: the participation of a delegation from the USA, sponsored by Committee A-1, in the activities of ISO/TC 17 on Steel; development of plans for the participation of the ASTM-ASME Joint Committee on Effect of Temperature on Metals in cooperation with the Metals Engineering Division of ASME and the Institution of Mechanical Engineers (Great Britain) in a joint International Conference on Creep and Fatigue to be held in New York in 1963; and the discussions between ASTM and the International Union of Testing and Research Laboratories for Materials and Construction (RILEM) in the development of an International Symposium on the Use of Atomic Physics for Testing Materials. This symposium may be held in conjunction with the 1962 Nuclear Congress in New York.

Long-Range Planning Activities

The Long-Range Planning Committee was established to determine whether, for the next decade, the Society organization and procedures are such that it can meet the challenges posed by industry and Government in the fields of research and standards for materials. LRP in its recommendations hopes to draw elements in the Society closer together and to have ASTM organized so that there will be a minimum of delays in handling problems that are developing so rapidly in the new technology.

In undertaking its studies, LRP drafted some 60 broad questions, answers to which should indicate what changes and additions need to be made so that ASTM can best meet the challenges ahead. Among the major problems and subjects being covered by LRP are:

1. ASTM Name, Scope, Purposes, and Services,
2. Technical Committees,
3. Membership Promotion and Aspects,
4. Meetings: National, District, Committee; and Meeting Programs,
5. Standardization Procedures,
6. Research Activities,
7. Publications,
8. Inter-Society Relations,
9. Government Relations: Federal, State, Municipal,
10. Promotional and Development, Publicity,
11. University and Technical Schools,
12. American Standards Assn.; International Standardization,
13. Membership Appraisal,
14. Board of Directors: Terms; Membership; Operations,
15. Headquarters Staff Organization,
16. Branch Offices,
17. Location of Headquarters Offices and Facilities, and
18. Finances.

To get a broad consensus of membership opinion, several subcommittees and special task groups were organized. Since its first meeting there have been some 30 meetings of the main LRP and the subdivisions. A complete list of meetings and subgroups will appear in the final report. Meanwhile, many members with long service and widespread experience, not only in ASTM, but also in the work of other societies, have been devoting many hours in evaluating questions and problems.

Based on recommendations from LRP, a number of actions have already been taken: Membership of the Board of Directors was increased; an annual doctoral fellowship award of $6500 was established; five annual grants-in-aid of $1000 each for work in materials are being made; the Materials Sciences Division, which is expected to become a very important part of ASTM operations, stemmed from LRP recommendations.

While widespread publicity has not been given to the LRP work in some areas, the membership has been asked to comment, for example, on the desirability of a change in ASTM's name to indicate better its broad scope with respect to materials.

All of the suggestions are by no means affirmative. For example, a special task group of experts in the field of information retrieval functioning under the LRP Study Group on Publications has recommended that ASTM not sponsor a special symposium or sessions dealing with methods of information storage, evaluation, and retrieval. It is their belief that solutions to this problem are still in the formative stages and such a symposium would only be repetitive of material already discussed and published under other auspices. Meanwhile, however, a review paper for the information of ASTM members is being prepared for publication in *Materials Research & Standards*.

At its May meeting the Board received final reports from LRP dealing with Membership and Membership Promotion, Inter-Society Relations, and Developmental Activities, and in addition a partial recommendation in the publications area. The membership report proposes a more aggressive stimulation of the Society's membership because of the very sound and worthwhile services the Society has to offer its members, both individual and company. In the field of inter-society relations, the nature of ASTM activity, embracing as it does the whole field of materials, justifies the development of more cooperative and helpful relations with other organizations, particularly professional, scientific, and trade organizations.

The Board has been apprised of progress in several other important areas under study by LRP. Two subcommittees, one on Standardization Activities and Procedures under the chairmanship of Albert T. Goldbeck, and the other on Technical Committee Activities under the chairmanship of ASTM Vice-President R. Wade Seniff, have been struggling with the important problem of improving our standardization procedures and our technical committee operations. In addition, the main committee itself has been studying the very important problem of the publication program of the Book of Standards which because of its growth has created problems in several areas, including printing, editing, and sales.

The wisdom and knowledge of the many members and friends of the Society participating in the LRP studies insures that there will be a thorough appraisal of almost every facet of ASTM operations. The main committee guiding these numerous studies and responsible for the recommendations being prepared for the Board of Directors is composed of the following current or former national officers of the Society:

Norman L. Mochel, Chairman
A. A. Bates
M. N. Clair
H. M. Hancock
Richard T. Kropf
F. L. LaQue
R. A. Schatzel
R. W. Seniff
K. B. Woods

Robert J. Painter, formerly Executive Secretary and Treasurer, now Consultant to the Executive Secretary, is devoting a major portion of his time to assisting Mr. Mochel and LRP in their work.

Respectfully submitted on behalf of the Board of Directors,

A. ALLAN BATES,
President.

T. A. MARSHALL, JR.,
Executive Secretary.

June, 1961.

APPENDIX I

REPORT OF THE AUDITORS FOR THE FISCAL YEAR DECEMBER 1, 1959 TO NOVEMBER 30, 1960

Philadelphia, December 23, 1960

AMERICAN SOCIETY FOR TESTING MATERIALS

Philadelphia, Pa.

We have examined the accompanying balance sheet of the American Society for Testing Materials as of November 30, 1960 and the related statements of cash receipts and disbursements of general fund and building fund, statement of operations and surplus of the general fund, and statements of changes in special and designated funds for the period December 1, 1959 to November 30, 1960. Our examination was made in accordance with generally accepted auditing standards, and accordingly included such tests of the accounting records and such other auditing procedures as we considered necessary in the circumstances.

It has been the practice of the Society to maintain its accounting records on the general basis of cash receipts and disbursements with the exception that accounts receivable for publication sales and dues receivable from members are recorded, and certain provisions for estimated publication costs and other reserves are also recorded. In addition, the accounts receivable include total billings of orders in those instances in which only partial shipments had been made.

This method of keeping the accounts has its limitations in that financial statements prepared from the books do not reflect the Society's financial position at any given date, nor the results of its operations for any definite period as they would be determined from the application of generally accepted accounting principles. Accordingly we are unable to express an over-all opinion on the financial position of the Society at November 30, 1960, or the results of its operations for the period then ended. However, it is our opinion that cash, investments, fixed assets, building fund assets, and special and designated fund assets are presented fairly in the balance sheet, and that the statements of cash receipts and disbursements of, and changes in, the various funds are presented fairly and on a basis consistent with that of the preceding fiscal period.

Respectfully submitted,

ARTHUR YOUNG & COMPANY

BALANCE SHEET AS OF NOVEMBER 30, 1960

(Including Special and Designated Funds)

ASSETS

General Fund:				
Current:				
Cash			$169 325.77	
Investment Fund Participation (at cost) (market value $792 391.56)			686 316.31	
Accounts Receivable			111 793.75	
Loans Receivable (Building Fund)			120 325.00	
Travel Deposit			425.00	
Total Current Assets—General Fund			1 088 185.83	
Furniture and Fixtures (at Cost Less Accumulated Depreciation)			40 437.08	
Total Assets—General Fund				$1 128 622.91
Building Fund:				
Cash			5 773.16	
Temporary Advance to General Fund			45 000.00	
Investment Fund Participation (at cost) (market value $37 896.84)			32 822.90	
Land, Cost			138 459.42	
Building and Construction Costs		$342 659.22		
Less, Accumulated Depreciation		113 800.00	228 859.22	
Total Assets—Building Fund				450 914.70
Special and Designated Funds:				
Cash:				
Committee Funds		77 047.01		
ASTM Research Fund		13 859.25		
Dudley Medal and Marburg Lecture Fund	$82.74			
Richard L. Templin Award Fund	336.05			
Sam Tour Award Fund	441.69			
F. E. Richart Award Fund	166.90			
Total Medal and Lecture Funds, Cash		1 027.38	91 933.64	
Investment Fund Participation (at cost) (market value $272 967.60):				
Committee Funds		132 867.03		
ASTM Research Fund		89 779.60		
Dudley Medal and Marburg Lecture Fund		8 010.48		
Richard L. Templin Award Fund		2 563.36		
Sam Tour Award Fund		1 995.04		
F. E. Richart Award Fund		1 209.11	236 424.62	
Other Investments (at cost):				
Committee Funds (market value $117 015.63)		116 317.19		
ASTM Research Fund (market value $30 854.25)		10 074.63	126 391.82	
Total Assets—Special and Designated Funds				454 750.08
Total Assets				$2 034 287.69

LIABILITIES

General Fund:			
Current:			
Temporary Advance from Building Fund		$45 000.00	
Advance Collections		20 201.10	
Due Committees:			
Joint Committee on X-ray Diffraction		196 027.34	
Joint Symposium Nuclear Meeting		1 476.77	
Technical Assistant—C-1		111.41	
International Organization for Standardization		1 133.55	
Pennsylvania Sales Tax		332.79	
Provision for:			
1960 Proceedings		43 700.00	
1960 Book of Standards Supplements		70 800.00	
Total Current Liabilities—General Fund		378 782.96	
Reserves:			
Life Membership Fund	$4 549.94		
Publication Fund	16 841.80		
Book of Standards (1961)	200 000.00		
Executive Retirement	12 373.60		
Retirement Fund	36 462.76		
Depreciation of Investments	20 000.00		
Additional Cost of Replacement of Headquarters Building	35 000.00		
Special Annual Meeting Entertainment Account	2 661.64		
Grants—Educational Fund	11 500.00		
	339 389.74		
Surplus	410 450.21	749 839.95	
Total Liabilities and Surplus—General Fund			$1 128 622.91
Building Fund:			
Notes Payable—General Fund		120 325.00	
Contributions from Members	203 998.89		
Contributions from ASTM General Fund	124 764.57		
Accumulated Income, Net	1 826.24	330 589.70	
Total Building Fund			450 914.70
Special and Designated Funds:			
Committee Funds, Unexpended Balance		326 231.23	
ASTM Research Fund:			
Principal	105 854.23		
Income	7 859.25	113 713.48	
Dudley Medal and Marburg Lecture Fund:			
Principal	8 010.48		
Income	82.74	8 093.22	
Richard L. Templin Award Fund:			
Principal	2 563.36		
Income	336.05	2 899.41	
Sam Tour Award Fund:			
Principal	1 995.04		
Income	441.69	2 436.73	
F. E. Richart Award Fund:			
Principal	1 309.11		
Income	66.90	1 376.01	
Total Special and Designated Funds			454 750.08
Total Liabilities and Funds			$2 034 287.69

SPECIAL AND DESIGNATED FUNDS

AS OF NOVEMBER 30, 1960

COMMITTEE FUNDS

Committee A-1	On steel	$970.30
Committee A-5	On corrosion of iron and steel	431.43
Committee B-3	On corrosion of non-ferrous metals and alloys	2 404.45
Committee B-4	On metallic materials for electrical heating, electrical resistance, and electrical contacts	51.74
Committee B-5	On copper and copper alloys, cast and wrought	1.21
Committee B-6	On die-cast metals and alloys	3 651.84
Committee B-8	On electrodeposited metallic coatings	16.28
Committee C-1	On cementitious, ceramic, concrete, and masonry materials:	
	General funds	2 125.67
	Cement reference laboratory	(483.31)[a]
	Cement reference laboratory—inspection	3 401.86
Committee C-1 – C-9	Cement and concrete reference laboratory	6 111.28
Committee C-7	On lime	8.67
Committee C-9	On concrete and concrete aggregates	176.77
	Sanford E. Thompson Medal Award	1 143.51
Committee C-14	On glass and glass products	22.68
Committee C-15	On manufactured masonry units	613.74
Committee C-16	On thermal insulating materials	220.61
Committee C-17	Flower fund	18.68
Committee C-18	On natural building stones	160.04
Committee D-1	On paint, varnish, lacquer, and related products	1 385.09
Committee D-2	On petroleum products and lubricants	511.72
	ASTM—IBM	1 179.77
	DCC I Reference fuel account	45 186.63
Committee D-3	DCC II National exchange group	(3 170.35)
Committee D-4	On road and paving materials	1 118.84
Committee D-5	On coal and coke	2 439.16
Committee D-7	On wood	58.57
	Wood pole research fund	1 515.34
	Wood pole—Program No. 2	233.12
Committee D-9	On electrical insulating materials	2 670.59
Committee D-10	On shipping containers	19.79
Committee D-11	On rubber and rubber-like materials	592.46
	Rubber special	1 876.48
Committee D-12	On soaps and other detergents	1 076.12
Committee D-13	On textile materials	2 755.54
Committee D-14	On adhesives	70.99
Committee D-15	On engine antifreezes	24.37
Committee D-18	On soils for engineering purposes	119.61
Committee D-19	On industrial water	226.50
	Research project	6 222.55
Committee D-20	On plastics	1 048.41
Committee D-27	On electrical insulating liquids and gases	777.12
Committee E-2	On emission spectroscopy	340.63
Committee E-3	On chemical analysis of metals	199.74
Committee E-4	On metallography	43.86
Committee E-7	On nondestructive testing	7 816.52
Committee E-9	On fatigue	291.00
Committee E-14	On mass spectrometry	2 389.83
Committee F-1	On materials for electron tubes and semiconductor devices	217.01
ASA sectional committee on specifications for cast iron pipe and fittings—general funds		1 698.96
Chicago district		3 121.38
Cleveland district		137.23
Detroit district		2 378.07
Mississippi Valley district		774.59
New England district		2 841.49
New York district		7.10
Northern California district		4 816.88
Ohio Valley district		5.04
Pacific Northwest district		518.56
Philadelphia district		1 021.12

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[a] ( ) indicate red figures.

Philadelphia district special (annual meeting account)		3 720.60
Pittsburgh district		1 839.62
Southern California district		5 198.95
Washington district		226.85
Western New York – Ontario district		1 644.76
Advisory committee on corrosion		33 162.48
Administrative committee on end-use products		334.28
Joint committee on X-ray diffraction:		
Chemical analysis		147 113.99
Research fellowship		7 117.51
Joint committee on leather		2.71
Reserve—future West Coast meetings		8 264.60
		326 231.23
Accounted for as follows:		
Cash in bank and on hand	$77 047.01	
Investments (at cost) (market value $117 015.63):		
$20 000 US Treasury 2½s due 8/15/63	19 925.00	
20 000 US Treasury 2½s due 11/15/61	19 400.00	
75 000 US Treasury 4⅝ per cent due 5/15/65	76 992.19	
Investment fund participation	132 867.03	$326 231.23

ASTM RESEARCH FUND

Principal Account

	Total	*Invested*	*Uninvested Cash*
Balance, December 1, 1959	$100 673.95	97 673.95	$3 000.00
Transfer from General Fund	3 000.00		3 000.00
Transfer from Income—Gain on Investments	2 180.28	2 180.28	
Balance, November 30, 1960	105 854.23	99 854.23	6 000.00
Investment Fund Participation	89 779.60		
Other Investments:			
618 shares General Motors Co. (Common) (Note A) (market value $26 342.25)	7 258.63		
48 shares American Telephone & Telegraph (Note A) (market value $4 512.00)	2 816.00	99 854.23	

Income Account

Balance, December 1, 1959	4 077.70	
Receipts:		
Interest, Dividends and Gain on Investments	5 734.04	
Contributions—ASTM General Fund	2 000.00	11 811.74
Disbursements:		
Transfers to Principal	2 180.28	
Grants:		
Corrosion Research Council	500.00	
American Electroplaters Society	500.00	
Printing and Stationery	169.32	
Research—University of Delaware	602.89	3 952.49
Balance, November 30, 1960		$7 859.25

Note A.—The income from these securities is paid to annuitants.

Dudley Medal and Marburg Lecture Fund

Principal Account

Balance, December 1, 1959, Invested		$7 815.94
Transfer from Income—Gain on Investments		194.54
Balance, November 30, 1960, Invested		8 010.48

Income Account

Balance, December 1, 1959, Cash	$140.09	
Receipts:		
Interest, Dividends and Gain on Investments	511.62	
Transfers from General Fund	250.00	901.71
Disbursements:		
Transfer to Principal	194.54	
Dudley Medals	108.83	
Marburg Award	500.00	
Marburg Certificates	15.60	818.97
Balance, November 30, 1960, Cash		$82.74

Richard L. Templin Award Fund

Principal Account

Balance, December 1, 1959, Invested		$2 501.11
Transfer from Income—Gain on Investments		62.25
Balance, November 30, 1960, Invested		2 563.36

Income Account

Balance, December 1, 1959, Cash	$287.18	
Receipts:		
Interest, Dividends and Gain on Investments	163.72	450.90
Disbursements:		
Transfer to Principal	62.25	
Awards	50.00	
Certificates	2.60	114.85
Balance, November 30, 1960, Cash		$336.05

SAM TOUR AWARD FUND

Principal Account

Balance, December 1, 1959, Invested	$1 946.59
Transfer from Income—Gain on Investments	48.45
Balance, November 30, 1960, Invested	1 995.04

Income Account

Balance, December 1, 1959, Cash	$399.02	
Receipts:		
Interest, Dividends and Gain on Investments	127.42	526.44
Disbursements:		
Transfer to Principal	48.45	
Awards	35.00	
Certificates	1.30	84.75
Balance, November 30, 1960, Cash		$441.69

F. E. RICHART AWARD FUND

Principal Account

	Total	*Invested*	*Uninvested Cash*
Balance, December 1, 1959	$1 179.75	$1 179.75	
Contributions	100.00		$100.00
Transfer from Income—Gain on Investments	29.36	29.36	
Balance, November 30, 1960	$1 309.11	1 209.11	100.00

Income Account

Balance, December 1, 1959, Cash	134.64	
Receipts:		
Interest, Dividends and Gain on Investments	77.22	211.86
Disbursements:		
Transfer to Principal	29.36	
Award	100.00	
Certificates	15.60	144.96
Balance, November 30, 1960, Cash		$66.90

GENERAL FUNDS: CASH RECEIPTS AND DISBURSEMENTS

FOR THE PERIOD DECEMBER 1, 1959 TO NOVEMBER 30, 1960

Cash Balance, December 1, 1959			$136 569.41
RECEIPTS			
Operating Receipts (Budgeted):			
Dues and Entrance Fees:			
Current Dues	$371 902.88		
Past Dues	2 894.75		
Advance Dues	2 443.77		
Entrance Fees	7 641.90		
Income, Life Membership	175.00	$385 058.30	
Sales of Publications:			
Book of Standards (Members, Additional Parts)	100 036.06		
Book of Standards, 1955	996.40		
Book of Standards, 1958	175 642.97		
Supplement to Book of Standards, 1959	114 028.42		
Supplement to Book of Standards, 1960	2 979.60		
Methods of Chemical Analysis of Metals	4 422.15		
Compilations of Standards	140 854.55		
Separate Standards	64 144.93		
Selected Standards for Students	4 942.47		
Proceedings	8 261.30		
Bulletin Subscriptions (Nonmembers)	7 182.15		
Special and Miscellaneous Publications	249 610.58	873 101.58	
Miscellaneous:			
Advertising—BULLETIN	50 253.11		
Advertising—Index	4 213.10		
Interest and Dividends	28 675.42		
Registration and Other Fees—Meetings	35 315.00		
ASTM Exhibit at Annual Meeting	17 526.00		
Staff Service	17 855.15		
Furniture and Fixtures	15.00		
Miscellaneous	884.84	154 737.62	
Nonoperating Receipts (Not Budgeted):			
Excess Remittances	$4 472.85		
Committee C-1 for Technical Assistant	3 324.98		
Sale of X-Ray Diffraction Cards	139 551.80		
From Building Fund on Account of Loan	17 000.00		
Gain on Sales of Securities	16 667.08		
Gillett Memorial	566.57		
Loan from Investment Fund	20 000.00		
Loan from Building Fund	75 000.00		
San Francisco—Buffet Supper	185.00		
International Organization for Standardization Technical Committee 17—Meeting	2 685.59		
Annual Meeting—Luncheons	1 994.50		
Annual Meeting—Buffet	446.85		
Miscellaneous	89.60	281 984.82	
Total Receipts			1 694 882.32
Total Receipts for Period December 1, 1959 to November 30, 1960 and Cash Balance December 1, 1959			1 831 451.73

Brought forward			$1 831 451.73

DISBURSEMENTS

Operating Disbursements (*Budgeted*):			
Publications:			
Year Book	$18 408.28		
Book of Standards—1958	2 897.60		
Supplement to Book of Standards—1959	8 950.16		
Supplement to Book of Standards—1960	18 192.91		
Compilations of Standards	47 521.23		
Methods of Chemical Analyses of Metals	528.07		
Separate Standards	23 263.02		
Proceedings	3 284.12		
Preprints	15 453.10		
ASTM BULLETIN	68 216.28		
Circulars to Members	3 821.02		
Index to Standards	9 961.44		
Special and Miscellaneous and West Coast Publications	193 231.44	413 728.67	
Salaries	459 522.00		
General Office Expenses	131 038.90		
Expenses—Technical and District Committees	14 429.01		
Expenses—Meetings	18 026.27		
Headquarters Occupancy Expense (Includes $15 000 Depreciation on Buildings)	61 501.76		
American Standards Association	2 250.00		
Traveling Expense, Administrative and Special Committees	10 265.35		
Furniture and Fixtures	7 388.94		
Dues, Contributions, Miscellaneous	3 944.71		
Legal Services	2 000.00		
ASTM Educational Fund: Grants	11 500.00		
ASTM Research Fund	3 000.00		
Employees' Retirement Fund	46 595.73		
Group Insurance	4 714.44		
Blue Cross	2 757.60		
Major Medical	2 523.15		
Social Security	10 429.19	791 887.05	
Nonoperating Disbursements (*Not Budgeted*):			
Refund of Excess Remittances	4 472.85		
Technical Assistant Committee C-1	3 270.98		
Transfer to Joint Committee on X-Ray Diffraction	160 000.00		
Costs and Refunds X-Ray Diffraction Cards	42 286.99		
Executive Retirement Reserve	1 200.00		
Employee Retirement Reserve	290.64		
Book of Standards—1959 Supplement	81 000.00		
Proceedings—1959	40 000.00		
Investments	16 667.08		
Repayment of Load from Investment Fund	20 000.00		
Repayment of Bank Loan	50 000.00		
Repayment of Loan from Building Fund	30 000.00		
Gillett Memorial	571.22		
Annual Meeting—Luncheons	2 319.58		
Annual Meeting—Buffet	521.20		
Special Annual Meeting Account	1 000.00		
Staff Services	459.16		
Insurance	305.00		
Miscellaneous	593.50		
International Organization for Standardization Technical Committee 17—Meeting	1 552.04	456 510.24	
Total Disbursements			1 662 125.96
Cash Balance, November 30, 1960			$169 325.77

BUILDING FUND: ASSETS AND LIABILITIES

AS OF NOVEMBER 30, 1960

Assets

Cash—Girard Trust Corn Exchange Bank, Philadelphia, Pa.				$5 773.16
Investment Fund Participation (at cost)				32 822.90
Temporary Loan to General Fund				45 000.00
Properties (at cost):				
1916–18 Race Street, Philadelphia, Pa.:				
Land		$30 000.00		
Building	$26 503.35			
Construction (Net of Salvage)	138 690.83			
Additions 1960:				
Reinstallation of Cast Iron Railing	100.00			
	165 294.18			
Less, Accumulated Depreciation	77 783.05	87 511.13	$117 511.13	
1921–1927 Cherry Street, Philadelphia, Pa. and 1922–1928 Quarry Street, Philadelphia, Pa.:				
Land		30 800.00		
Building	38 880.10			
Construction	77 628.72			
	116 508.82			
Less, Accumulated Depreciation	26 416.95	90 091.87	120 891.87	
1921–23 Arch Street, Philadelphia, Pa. and 1920 Cherry Street, Philadelphia, Pa.:				
Land		70 000.00		
Buildings	59 830.75			
Additions 1960:				
Valuation Report	135.00			
	59 965 .75			
Less, Accumulated Depreciation	9 600.00	50 365.75	120 365.75	
1919 Cherry Street, Philadelphia, Pa.:				
Land		7 659.42		
Improvements		890.47	8 549.89	367 318.64
Total Assets				450 914.70

Liabilities and Funds

Loan Payable to ASTM General Fund		120 325.00	
Contributions from Members	203 998.89		
Contributions from ASTM General Fund	124 764.57		
Accumulated Income (Net)	1 826.24	330 589.70	$450 914.70

BUILDING FUND: CASH RECEIPTS AND DISBURSEMENTS

FOR THE PERIOD DECEMBER 1, 1959 TO NOVEMBER 30, 1960

Cash Balance, December 1, 1959		$32 578.77
Receipts		
Contributions from Members	$5 562.40	
Interest and Dividends on Investments	1 299.24	
Profit on Sale of Investments	797.10	
Rentals of 1920 Cherry Street	1 197.00	
From ASTM General Fund:		
On Account of Depreciation on Buildings, Elevator and Air Conditioning Installation	15 000.00	
On Account of Amortization of Note	17 000.00	
On Account of Temporary Advance	30 000.00	70 855.74
		103 434.51
Disbursements		
Cherry Street property:		
Real Estate Taxes 1960	2 931.33	
Arch Street property:		
Valuation Report on Building	135.00	
Real Estate Taxes 1960	1 597.92	
Race Street Property:		
Installation of Railing	100.00	
Reduction of Loan from ASTM General Fund	17 000.00	
Temporary Advance to ASTM General Fund	75 000.00	
Reimbursement of Contribution from Member	100.00	
Transferred to Investment Fund	797.10	97 661.35
Cash Balance, November 30, 1960		$5 773.16

ASTM INVESTMENT FUND

AS OF NOVEMBER 15, 1960

Cash	$9 815.29	
Investments (at cost) (Market value $1 093 440.71)	945 748.54	
Total Assets		$955 563.83
Participations by Funds		$955 563.83

PARTICIPATION BY FUNDS

	Participation November 16, 1959	*Net Profit, Sale of Securities Reinvested*	*Participation November 15, 1960*
General Fund	$669 649.23	$16 667.08	$686 316.31
Committee Funds	129 640.38	3 226.65	132 867.03
Research Fund	87 599.32	2 180.28	89 779.60
Building Fund	32 025.80	797.10	32 822.90
Medal and Lecture Fund	7 815.94	194.54	8 010.48
Templin Award Fund	2 501.11	62.25	2 563.36
Sam Tour Fund	1 946.59	48.45	1 995.04
Richart Fund	1 179.75	29.36	1 209.11
	$932 358.12	$23 205.71	$955 563.83

ASTM INVESTMENT FUND: CASH RECEIPTS AND DISBURSEMENTS

FOR THE PERIOD NOVEMBER 17, 1959 TO NOVEMBER 15, 1960

Cash Balance, November 17, 1959				$25 094.81
Receipts				
Interest and Dividends on Investment		$40 972.83		
Interest on Savings Accounts		101.42		
Total Interest and Dividends			$41 074.25	
Proceeds from Sales of Investments		217 713.15		
Reinvestment of Capital Gain by Participants		23 205.71		
Repayment of Loan to General Fund		20 000.00	260 918.86	
Total Receipts				301 993.11
				327 087.92
Disbursements				
Purchase of Securities			232 992.67	
Investment Counsel			3 250.00	
Loan to General Fund			20 000.00	
Distribution of Interest and Dividends and Gain to Participants:				
	Interest and Dividends	*Gain*		
General Fund	$27 166.58	16 667.08		
Committee Funds	5 259.29	3 226.65		
Research Fund	3 553.76	2 180.28		
Building Fund	1 299.24	797.10		
Medal and Lecture Fund	317.08	194.54		
Templin Award Fund	101.47	62.25		
Sam Tour Fund	78.97	48.45		
Richart Fund	47.86	29.36		
	$37 824.25	$23 205.71	61 029.96	
Total Disbursements				317 272.63
Cash Balance, November 15, 1960				9 815.29
Balance Composed of:				
Demand Account			6 763.27	
Savings Account			3 052.02	$9 815.29

INVESTMENT FUND: LIST OF SECURITIES

AS OF NOVEMBER 15, 1960

Investments

Number of Shares or Par Value	*Bonds*	*Cost or Book Value*	*Market Value*
$10 000	Commercial Credit Co. 4½ per cent 3/1/77	$9 806.00	$9 300.00
20 000	Consolidated Natural Gas Co. Deb. 5s 2/1/85	20 383.50	20 100.00
8 000	Consumers Power Co. 1st 4¾ per cent 10/1/87	8 100.00	8 100.00
20 000	General Motors Acceptance Corp. 3½ per cent Deb. 3/15/72	18 250.00	17 875.00
15 000	General Motors Corp. 3¼ per cent Deb. 1/1/79	14 925.00	13 500.00
20 000	Scott Paper Co. 3 per cent Conv. Deb. 3/1/71	20 377.80	22 350.00
15 000	Southern Natural Gas Co. 1st Mtg Pipe Line S F 3⅛ per cent 2/1/74	14 734.81	12 750.00
15 000	Southwestern Bell Telephone Co. 4¾ per cent Debs. 10/1/92	15 450.00	14 550.00
15 000	Superior Oil Co. 3¾ per cent Deb. 7/1/81	15 000.00	13 668.75
10 000	Tennessee Gas Transmission Co. 1st Mtg Pipe Line 4¾s 11/1/76	10 065.00	9 850.00
10 000	U. S. Federal Land Banks 4 per cent 9/20/61	10 027.00	10 062.50
10 000	Federal Land Banks 3⅞ per cent 9/15/72	9 806.25	9 650.00
15 000	Twelve Federal Land Banks 4⅛ per cent 2/15/72–67	15 000.00	14 850.00
15 000	Federal National Mortgage Association 4⅛ per cent 11/12/63	15 002.00	15 168.75
15 000	U. S. Treasury 2½s 6/15/67–72	14 694.08	13 059.38
50 000	U. S. Treasury 2⅝s 2/15/65	45 722.75	47 937.50
15 000	U. S. Treasury 2¾s 9/15/61	14 934.38	14 976.56
25 000	U. S. Treasury 3⅝s 5/15/61	24 906.25	25 085.94
20 000	U. S. Treasury 3s 8/15/66	18 977.00	19 387.50
15 000	U. S. Treasury 3¾ per cent 5/15/64	15 000.00	15 051.56
20 000	U. S. Treasury 4s 8/1/61	20 056.25	20 143.75
45 000	U. S. Treasury 4⅝s 5/15/65	45 000.00	46 532.81
25 000	U. S. Treasury 4⅞s 11/15/63	25 000.00	25 929.69
15 000	U. S. Treasury 5s 8/15/65	15 000.00	15 665.63
	Total Bonds	436 218.07	435 545.32

Preferred Stocks

Shares			
30	Endicott Johnson Corp. 4 per cent Cum Pfd	$2 937.50	$2 400.00
30	General Motors Corp. $5 Cum Pfd	3 755.80	3 142.50
30	Long Island Lighting Co. 4.35 per cent Series E Cum Pfd	3 015.00	2 610.00
30	P. Lorillard Co. 7 per cent Cum Pfd	4 380.00	4 080.00
30	Mississippi Power & Light Co. 4.36 per cent Cum Pfd	3 075.00	2 580.00
100	Philadelphia Electric Co. 4.4 per cent Cum Pfd	10 670.00	9 850.00
47	Standard Slag Co. 2nd Pfd	2 115.00	2 115.00[a]
30	Westinghouse Electric Corp. 3.80 per cent Cum Pfd Series B	3 067.74	2 610.00
	Total Preferred Stocks	33 016.04	29 387.50

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[a] Market value not available.

Shares	*Common Stocks*		
150	Abbott Laboratories	$8 884.37	$8 400.00
202	Allegheny Ludlum Steel Corp.	8 111.87	7 272.00
100	Aluminum Co. of America	2 061.45	6 825.00
200	Aluminium, Ltd.	6 922.26	6 025.00
100	Amerada Petroleum Corp.	10 399.35	6 850.00
250	American Can Co.	10 395.00	8 250.00
194	American Stores Co.	7 769.21	12 731.25
300	American Telephone & Telegraph Co.	17 465.36	28 200.00
160	Bethlehem Steel Corp.	2 095.99	6 440.00
139	Chase Manhattan Bank	3 947.42	7 992.50
200	Cleveland Electric Illuminating Co.	8 271.00	9 900.00
300	Consolidation Coal Co.	12 071.32	9 150.00
100	Crown Zellerbach Corp.	2 914.79	4 662.50
200	Cutler-Hammer, Inc.	16 920.33	15 700.00
179	Dow Chemical Co.	4 125.15	14 118.63
50	E. I. duPont de Nemours & Co.	6 544.18	9 700.00
200	Emhart Manufacturing Co.	10 938.85	11 000.00
400	Federated Department Stores, Inc.	14 007.82	14 800.00
125	General Electric Co.	4 311.15	9 812.50
450	General Mills, Inc.	10 630.89	13 162.50
675	General Telephone & Electronics Corp.	3 312.82	18 393.75
400	Goodyear Tire & Rubber Co.	14 020.38	15 600.00
150	Great Northern Paper Co.	12 688.34	6 450.00
458	Gulf Oil Corp.	3 566.31	13 568.25
238	Houston Lighting & Power Co.—Texas	8 061.31	19 992.00
200	Jewel Tea Co.	9 686.01	9 400.00
200	Johns-Manville Corp.	8 063.78	10 875.00
107	Jones Laughlin Steel Corp.	2 670.72	5 898.38
100	Mead, Johnson & Co.	7 621.83	12 850.00
100	Merck & Co., Inc.	3 887.50	8 125.00
50	National Lead Co.	5 059.25	4 362.50
200	Northern Illinois Gas Co.	8 467.44	7 900.00
100	Northern Indiana Public Service Co.	6 295.25	6 300.00
200	Ohio Oil Co.	2 623.35	7 150.00
300	J. C. Penny Co.	12 644.63	12 750.00
250	Peoples' Gas, Light & Coke	13 632.36	15 687.50
337	Philadelphia Electric Co.	9 327.20	17 187.00
105	Philips' Gloeilampenfabrieken	8 847.80	16 800.00
500	Potomac Electric Power Co.	13 122.32	14 875.00
203	Radio Corporation of America	11 025.30	11 088.88
200	Republic Steel Corp.	8 523.26	11 050.00
200	Sherwin-Williams Co.	10 515.69	23 100.00
138	Socony Mobil Oil Co.	3 103.03	5 364.75
100	Southern California Edison Co.	6 558.01	6 100.00
150	Southern Railway Co.	5 998.81	6 900.00
156	Square D Co.	2 362.37	4 660.50
200	Standard Oil Co. of Indiana	11 969.64	8 850.00
288	Standard Oil Co. of New Jersey	8 971.97	11 664.00
200	Sterling Drug, Inc.	12 565.48	12 300.00
450	Tennessee Gas Transmission Co.	10 483.88	10 350.00
288	Texaco, Inc.	14 195.17	22 608.00
60	Union Carbide Corp	4 235.24	7 170.00
200	The Upjohn Co.	10 538.45	9 975.00
200	Western Union Telegraph Co.	9 359.38	7 950.00
480	Westinghouse Electric Corp.	15 644.26	24 480.00
242	Wisconsin Electric Power Co.	8 078.13	9 740.50
	Total Common Stocks	476 514.43	628 507.89

Summary

Bonds	436 218.07	435 545.32
Preferred Stocks	33 016.04	29 387.50
Common Stocks	476 514.43	628 507.89
	$945 748.54	$1 093 440.71

APPENDIX II

REPORT OF ADMINISTRATIVE COMMITTEE ON STANDARDS

The Administrative Committee on Standards is responsible to the Board of Directors for the supervision of the standardization activities of the Society. In carrying out this responsibility during the past year, the administrative committee has actively concerned itself in the following areas of standardization:

1. Promotion and expansion of standardization work in new fields,

2. Formulation of a general policy concerning standardization activities,

3. Review of the Society's standardization work, and

4. Action on recommendations from technical committees on proposed new standards and proposed amendments of existing tentatives or of existing standards offered between Annual Meetings of the Society.

These activities of the Administrative Committee on Standards are described in more detail later in this report, but the more important actions taken in the several categories are referred to briefly here.

In the area of promotion and expansion of standardization work, the administrative committee took action on the following: (1) Ceramics for Electronics, (2) Sensory Evaluation of Materials and Products, (3) Methods for Analysis of Radioisotopes, (4) Skid Resistance, (5) Gas Chromatography, and (6) Slide Fasteners.

The administrative committee has given serious consideration to several matters having to do with general policy concerning standardization activities:

1. Multiple sponsorship of sectional committees functioning under ASA Procedure,

2. A new procedure in lieu of the present annual meeting procedure in acting upon recommendations from the technical committees, and

3. The establishment of a policy with respect to detailed descriptions of apparatus used in ASTM methods of testing.

Actions by the Society at 1960 Annual Meeting:

In the area of review of the Society's standardization work, the administrative committee took cognizance of the recommendations affecting standards accepted by the Society at the 1960 Annual Meeting. The various actions reported are:

New Standards and Existing Tentatives Adopted as Standard	151
Standards in Which Revisions Were Adopted	102
New Tentatives	129
Standards Revised and Reverted to Tentative	17
Tentative Revisions of Standards	12
Tentatives Revised	141
Standards and Tentatives Withdrawn	12

Actions by the Administrative Committee on Standards During the Year:

It is encouraging to note that an increasing number of the technical committees are taking advantage of the procedure of the Administrative Committee on Standards for action on recommendations between annual meetings. During the past year, no less than 28 technical committees submitted a total of 130 proposals for review and action as follows:

New Tentatives	56
Revisions of Tentatives	46
Standards Revised and Reverted to Tentative	22
Tentative Revisions of Standards	5
Tentative Withdrawn	1

In addition, reports from seven sectional committees, for which ASTM serves as sponsor, were reviewed as referred to in a later section of this report.

The Administrative Committee on Standards carries on the bulk of its activities through correspondence. Since the last Annual Meeting of the Society, the committee held one meeting on Sept. 29, 1960, at Society Headquarters.

An election of officers was held at the September meeting at which time G. H. Harnden was elected chairman. The retiring chairman, J. H. Foote, had served for a period of seven years and the committee wishes to record its sincere appreciation of his interest and devoted service during these years.

EXPANSION OF STANDARDIZATION ACTIVITIES

A number of new standardization projects are discussed below:

Skid Resistance.—Mention was made in the report a year ago of considerations that had developed as a result of a request from the Highway Research Board that the Society undertake work in the field of methods of testing that would be helpful in connection with the problem of skid resistance. In the light of these discussions, the Board authorized a new Committee E-17 on Skid Resistance which held its organization meeting during the Annual Meeting in 1960. While the original inquiry had in mind primarily skid resistance of highways, the scope of the new committee is sufficiently broad to encompass all types of traffic surfaces.

Ceramics for Electronics.—The 1960 report referred to a new Committee C-25 on Ceramics for Electronics that had been authorized as a result of a request to establish a committee to work with the newly formed Electronics Division that had been organized by the American Ceramic Society. The organization meeting of this committee was held on April 28, 1960.

Sensory Evaluation of Materials and Products.—Under the heading "Psychophysical Testing" the report a year ago gave some of the history leading up to the organization of a new Committee E-18 on Sensory Evaluation of Materials and Products. The organization meeting of the committee was held on Oct. 12, 1960. This committee will serve as the focal point for discussions in which a number of ASTM technical committees are concerned since many of these committees are faced with this type of evaluation in connection with such properties as odor and taste that may be transmitted to products in which the materials under the jurisdiction of these committees come in contact.

Gas Chromatography.—Seldom has such enthusiasm been shown in connection with the organization of any work by the Society as that which developed with respect to the organization of the new Committee E-19 on Gas Chromatography. Several of the Society's committees had already started work on specific methods in this field, but when Committee E-15 on Analysis and Testing of Industrial Chemicals took steps to organize a new subcommittee to deal with the general aspects of such methods of testing, it developed that the interest was so widespread that a new technical committee should be established, which was promptly authorized by the Board.

Over 100 potential members attended the organization meeting held on Feb. 6, 1961, and the subcommittee organization is already functioning.

Methods for Analysis of Radioisotopes.—Upon looking into the question of undertaking work on methods of analysis of radioisotopes, it developed that this would be an appropriate subject for Committee E-10 on Radioisotopes and Radiation Effects, and this committee has accepted jurisdiction for this work. In this the committee will have the cooperation of Committee E-3 on Chemical Analysis of Metals and of Committee E-15 on Analysis and Testing of Industrial Chemicals which had been making inquiry concerning the subject.

Slide Fasteners.—One of the projects requiring early attention in the estimation of the Subcommittee on Ultimate Consumer Textile Products of Committee D-13 on Textile Materials was that of evaluating slide fasteners. Complete methods were developed in the committee, but since engineering design is involved to some degree the subject was brought to the attention of the Board of Directors. The Board authorized this work to proceed subject to review by the Standards Committee. The committee has approved the submission of these standards to the Society for publication as tentative.

Gall and Wear Testing.—Although considerable interest had been expressed in having the Society undertake work in the field of gall and wear testing, no progress can be reported up to this time with respect to any further developments.

End Products.—More and more the Society is being approached for specifications covering end products. One of these requests dealt with plastic products for use in the building industry and was brought to the Society's attention by the Building Research Advisory Board. Similarly, requests have been received in connection with aluminum guard rails for highways, and more recently specifications in the glass field such as glass piping. The Society's Committee C-14 on Glass and Glass Products is being looked to to explore the possibilities in this field.

RELATIONS WITH AMERICAN STANDARDS ASSN.

Two methods under the procedure of the American Standards Association are used by ASTM in submitting recommendations to the ASA. One is a combination of the so-called existing standards procedure and the proprietary sponsorship procedure. On the initial reference of an existing ASTM standard to the ASA, it is submitted under the existing standards procedure with a supporting statement giving a history of the development of the standard and indicating the degree of its acceptance. If the standard is approved as American Standard, the ASTM is granted proprietary sponsorship insofar as any future revisions are concerned, and such revisions are brought to the attention of the ASA currently for approval under the proprietary sponsorship procedure.

The second method consists of having standards reviewed in a sectional committee made up of representatives of a number of interested organizations, usually with the ASTM as sponsor or co-sponsor of the sectional committee.

Standards Submitted Under the Existing Standards Procedure:

No standards were submitted to the ASA during the year under the existing standards procedure.

Revised Standards Submitted Under the Proprietary Procedure:

In view of the Society having been designated as proprietary sponsor for the revision of ASTM standards that had been approved as American Standard under the existing standards procedure, it has submitted to the American Standards Association during the year revisions of 3 standards relating to steel, 1 relating to corrosion of iron and steel, 1 relating to copper and copper alloys, 1 relating to electrodeposited metallic coatings, 1 relating to refractories, 3 to gypsum, 3 to manufactured masonry units, 1 to wood, 3 to bituminous materials for waterproofing, 1 to soap, 1 to plastics, and 1 to thermometers.

Standards Submitted Under the Sectional Committee Procedure:

During the year, the following sectional committees, for which the Society is sponsor, took action to present recommendations to the ASA as follows:

Sectional Committee A1 on Hydraulic Cements.—This committee has recommended the approval as American Standard of 8 standards, the approval of revisions of 9 American Standards, and the reaffirmation of 1 American Standard.

Sectional Committee B36 on Wrought-Iron and Wrought-Steel Pipe and Tubing.—This committee has recommended the approval of revisions of 14 American Standards.

Sectional Committee C7 on Bare Electrical Conductors.—This committee has recommended the approval as American Standard of 3 ASTM standards, the approval of revisions of 11 American Standards, and the reaffirmation of 2 American Standards.

Sectional Committee C59 on Electrical Insulating Materials.—This committee has recommended the approval of revisions of 2 American Standards and the withdrawal of 1 American Standard.

Sectional Committee L14 on Textile Test Methods.—This committee has recommended the approval of revisions of 6 American Standards and the withdrawal of 3 American Standards.

Sectional Committee Z11 on Petroleum Products and Lubricants.—This committee has recommended the approval as American Standard of 3 ASTM standards, the approval of revisions of 9 American Standards, and the reaffirmation of 35 American Standards.

Sectional Committee Z23 on Specifications for Sieves for Testing Purposes.—This committee has recommended the approval of revisions of 1 American Standard.

INTERNATIONAL RELATIONS

The international discussions, particularly under the International Organization for Standardization (ISO) and the International Electrotechnical Commission (IEC), are assuming ever greater significance. A number of the European countries apparently wish to have the standardization discussions handled internationally and much of the work is supported here rather than in the development of individual national standards. In certain areas these international discussions are having a definite impact on the standardization in this country, and a number of ASTM committees are following these international discussions quite closely with the thought that the ASTM standards and the international recommendations should be in harmony. A list of the various ISO and IEC projects in which ASTM is interested and which continues to grow year by year is given below. Reports appear currently in *Materials Research & Standards* in connection with meetings of these ISO committees, which refer to the actions taken and to the delegations that attend these meetings.

ISO Committees:

Aromatic Hydrocarbons.—ASTM Committee D-16 on Industrial Aromatic Hydrocarbons and Related Material is handling participation by the United States in the work of ISO/TC 78 on

Aromatic Hydrocarbons for which Great Britain holds the secretariat.

Asbestos - Cement Products.—ASTM Committee C-17 on Asbestos-Cement Products is handling participation by the United States in the work of ISO/TC 77 on Asbestos-Cement Products for which Switzerland holds the secretariat.

Cast Iron.—Great Britain holds the secretariat for ISO/TC 25 on Cast Iron. The Advisory Committee of ASTM Committee A-3 on Cast Iron has been designated to handle participation by the United States.

Chemistry.—Italy holds the secretariat for ISO/TC 47 on Chemistry. The United States recently has become a participating member of this committee so far as test methods are concerned, and ASTM Committee E-15 on Analysis and Testing of Industrial Chemicals will handle United States participation.

Copper and Copper Alloys.—The United States found it necessary to relinquish the secretariat for ISO/TC 26 on Copper and Copper Alloys but it will continue to participate in the work through the Advisory Committee of ASTM Committee B-5 on Copper and Copper Alloys, which has been designated as the American Group for this ISO work.

Fibre Building Board.—ASTM Committee D-7 on Wood has recommended that United States participation in the work of ISO/TC 89 on Fibre Building Board be limited to that of observer. Germany holds the secretariat for ISO/TC 89.

Fire Tests for Building Materials and Structures.—ASTM Committee E-5 on Fire Tests of Materials and Construction has recommended against United States participation in the work of ISO/TC 92 on Fire Tests for Building Materials and Structures on the basis that such participation could not be justified in view of the expense involved.

Hydraulic Binders.—ASTM Committee C-1 on Cement is being looked to to be responsible for the technical participation on the part of the United States in the work of ISO/TC 74 on Hydraulic Binders for which Belgium holds the secretariat.

Iron and Steel.—The United States is now participating in the work of ISO/TC 17 on Steel and the group to handle this participation has been set up under Committee A-1 on steel. Great Britain holds the secretariat for ISO/TC 17.

Laboratory Glassware.—Great Britain holds the secretariat for ISO/TC 48 on Laboratory Glassware. The United States is participating in this work through a special group on which ASTM is represented through Committee E-1 on Methods of Testing.

Light Metals and Alloys.—The Advisory Committee of ASTM Committee B-7 on Light Metals and Alloys has been designated to handle American participation in the work of ISO/TC 79 on Light Metals and Alloys. France holds the secretariat for this ISO committee.

Mica.—Subcommittee IX on Mica of ASTM Committee D-9 on Electrical Insulating Materials has been set up to handle the contacts by the United States with ISO/TC 56 on Mica for which India holds the secretariat.

Paper.—Participation by the United States in the work of ISO/TC 6 on Paper is being handled by a special committee composed of representatives of ASTM, the Technical Association of the Pulp and Paper Industry, and other interested groups. France holds the secretariat for this ISO committee.

Petroleum.—The United States holds the secretariat for ISO/TC 28 on Petroleum Products, the technical work of the secretariat being under the purview of Sectional Committee Z11 on Petroleum Products and Lubricants, for which ASTM is sponsor.

Plastics.—The United States holds the secretariat for ISO/TC 61 on Plastics.

ASTM Committee D-20 on Plastics is handling the work of the secretariat and has organized for the purpose a special committee consisting of representatives of the various groups interested.

Raw Materials for Paints, Varnishes, and Similar Products.—ASTM Committee D-1 on Paint, Varnish, Lacquer and Related Products is handling United States contacts with ISO/TC 35 on Raw Materials for Paints, Varnishes, and Similar Products, for which The Netherlands holds the secretariat.

Refractories.—ASTM Committee C-8 on Refractories is handling United States contacts with ISO/TC 33 on Refractories for which Great Britain holds the secretariat.

Rubber.—The secretariat for ISO/TC 45 on Rubber is held by Great Britain. The American Group for participation in this ISO committee has been set up under ASTM Committee D-11 on Rubber and Rubber-like Materials.

Shellac.—Subcommittee XIII on Shellac of ASTM Committee D-1 on Paint, Varnish, Lacquer and Related Products has been set up as the American committee in charge of contacts with ISO/TC 50 on Lac for which India holds the secretariat.

Shipbuilding Details.—Consideration is being given to the matter of participation by the United States in the work of ISO/TC 8 on Shipbuilding Details.

Sieves.—Sectional Committee Z23, for which the Society is co-sponsor with the National Bureau of Standards, handles the contacts for the United States in the work of ISO/TC 24 on Sieves.

Solid Mineral Fuels.—ASTM Committee D-5 on Coal and Coke is handling the contacts of the United States with ISO/TC 27 on Solid Mineral Fuels for which Great Britain holds the secretariat.

Surface Active Agents.—ASTM Committee D-12 on Soaps and Other Detergents has recommended participation by the United States in the work of ISO/TC 91 on Surface Active Agents. This has, however, not yet been cleared in the American Standards Assn., particularly as to organization of the United States group to handle this participation.

Statistical Treatment of Series of Observations.—The Netherlands holds the secretariat for ISO/TC 69 on Statistical Treatment of Series of Observations. The United States is participating in this work through a special group on which ASTM is represented.

Textiles.—Great Britain holds the general secretariat for ISO/TC 38 on Textiles, but the United States holds the secretariat for several subcommittees. Participation by the United States in this work is being handled by a special committee organized under the sponsorship of ASTM and the American Association of Textile Chemists and Colorists with representation from the groups interested. This special committee has been given the designation L23.

Viscosity.—The United States holds the secretariat for ISO/TC 66 on Viscosity. The Subcommittee on Rheological Properties of ASTM Committee E-1 on Methods of Testing has been set up to handle the work of the secretariat.

Zinc.—The United States has observer status in the work of ISO/TC 18 on Zinc for which Belgium holds the secretariat.

IEC Committees:

Insulating Oils.—E. R. Thomas, chairman of ASTM Committee D-27 on Electrical Insulating Liquids and Gases, is the technical advisor to the U. S. National Committee of the International Electrotechnical Commission in connection with the work of IEC/TC 10 on Insulating Oils.

Insulating Materials.—Arnold H. Scott, chairman of ASTM Committee D-9 on Electrical Insulating Materials, is technical advisor to the U. S. National Committee of the IEC for Committee 15 on Insulating Materials.

Magnetic Steel.—The technical advisor to the U. S. National Committee of the IEC for the work of Committee 14A on Magnetic Steel is A. C. Beiler, chairman of ASTM Committee A-6 on Magnetic Properties.

Aluminum.—E. J. Holcomb of the Aluminum Company of America is technical advisor to the U. S. National Committee of the IEC for Committee 7 on Aluminum. Mr. Holcomb looks to Sectional Committee C7, for which ASTM is sponsor, for assistance in processing recommendations from this IEC committee.

Respectfully submitted on behalf of the committee,

G. H. HARNDEN,
Chairman.

R. E. HESS,
Secretary.

APPENDIX III

REPORT OF ADMINISTRATIVE COMMITTEE ON RESEARCH

The Administrative Committee on Research met twice during 1960—on June 26 in Atlantic City, N. J., and on December 15 at ASTM Headquarters.

There are now eight committee members. F. T. Sisco and A. T. McPherson retired from the committee and W. F. Roeser and J. C. Harris have become new members.

Mr. B. W. Gonser, who has served so ably as chairman for six years, is planning to retire from the committee this year. Mr. W. J. Smith was elected the new chairman and E. I. Shobert was elected vice-chairman.

The research fund has continued its growth and now has a balance of approximately $106,000. Disbursements for research projects during the year have totaled about $8000. Of this amount, $6000 was in support of a Committee D-19 project on vaporous carryover of boiler water salts. Work on the project is now completed and a final report is planned for presentation at the 1961 Annual Meeting. Allocation of $1000 has been made to continue, through committee D-9 on Electrical Insulating Materials, ASTM participation in a research project on printed wiring at the University of Delaware during 1961.

The committee has continued its regular review of the various research projects in which ASTM has interest and participation.

Committee members were responsible for writing or providing short papers for publication in *Materials Research & Standards*. These appear as a feature under ACR Notes. Comments relative to them have been favorable and we believe that these informative and thought-provoking articles represent a very worthwhile contribution of the committee.

An important part of the committee effort is in seeking new ways to encourage and improve research attitudes in ASTM activities and to promote the knowledge of materials resulting from research. Recognizing that future progress will be guided importantly by those who are now the younger people, ways are being sought to stimulate greater participation of talented young scientists and engineers in the activities of professional societies. Under current consideration, also, is the possibility of promoting informal forums where research in progress can be reported and discussed without the need to prepare or publish the material involved.

It was voted by the committee that the chairman would act as its representative on the Council, Division of Materials Sciences.

Respectfully submitted on behalf of the committee,

WALTER J. SMITH,
Chairman.

FRANK Y. SPEIGHT,
Secretary.

APPENDIX IV

REPORT OF ADMINISTRATIVE COMMITTEE ON PAPERS AND PUBLICATIONS

The Administrative Committee on Papers and Publications held two meetings during the year, one on November 17, 1960, and the other on February 10, 1961.

Messrs. Robert Burns, National Research Council, and F. M. Howell, Aluminum Co. of America, have retired after serving two 3-year terms. Messrs. R. W. Fenn, Jr., The Dow Metal Products Co., and W. F. Simmons, Battelle Memorial Inst., were appointed for a 3-year term.

The publication program of the Society continues to expand. Apart from the Annual Meeting, which each year results in more special technical publications, and the triennial Pacific Area National Meetings, special symposia are being sponsored by technical committees which merit publication. For example, Committee D-2 on Petroleum Products and Lubricants sponsored a Symposium on Non-Newtonian Viscometry at its October meeting comprising two sessions, and the Society is being asked to publish this material. At its February meeting, Committee D-2 sponsored a very timely discussion on Current Research on Motor Gasoline Which May Affect Future Specifications, and the papers will be available at a very early date as *ASTM STP No. 298*. An innovation that may be establishing a worthwhile precedent is the holding of special national meetings, such as that held in Philadelphia, April 5 to 7, 1961. The technical program was organized by Committee F-1 on Materials for Electron Tubes and Semiconductor Devices and comprised a three-day Symposium on Materials and Electron Device Processing. The symposium dealt with the so-called ultra-clean rooms for processing of electronic materials and components as well as means for attaining and evaluating surface cleanliness of materials being processed. The Society is being asked to make these papers available promptly as *ASTM STP No. 300*, and these should result in a very valuable publication of some considerable size.

The review of the papers alone from these various meetings has resulted in a very busy year for the Committee on Papers and Publications, but in addition consideration was given to a number of important developments in connection with the Society's publications, as indicated below.

ASTM Bulletin:

Mention was made in the report a year ago of the committee's recommendation that the ASTM BULLETIN appear monthly rather than eight times a year as heretofore. During the year, consideration was also given to changing the cover and format with a change in name. With the approval of the Board of Directors, these changes became effective as of January 1 so that the BULLETIN will now appear monthly as *Materials Research & Standards*. This will provide an opportunity for more timely communication of news to the members and also lend emphasis to the technical nature of the papers published. The reception accorded these changes has been quite favorable. It is expected that the modifications will be attractive to advertisers so that, with the increased support, more material can be made available to the members.

Third Pacific Area National Meeting:

The Third Pacific Area National Meeting held in 1959, the largest ever sponsored by the Society, with 52 sessions and over 230 papers, has resulted in eighteen special technical publications. In addition some of the papers have appeared in the ASTM BULLETIN, now *Materials Research & Standards*.

Book of Standards:

Certain discussion now taking place in the Long-Range Planning Committee may result in some rather far-reaching changes in the method of publishing the Book of Standards (and possibly other publications). Staff studies are now going forward with respect to the advisability of having the Book of Standards issued in a greater number of parts corresponding in general to the special compilations that are now published in addition to the Book of Standards. With certain changes in concept concerning the propriety of including certain material which is now published in these compilations in addition to the standards, it is quite possible that by augmenting the special compilations now issued and with other adjustments they could constitute the Book of Standards.

The incompleteness of the present book was indicated in the analysis on adjuncts to the Book of Standards referred to in the report of the committee a year ago.

1961 Annual Meeting:

A full program is planned for the 1961 Annual Meeting consisting of 41 technical sessions; of these 16 will be devoted to special symposiums. The 9 symposiums making up these 16 sessions are listed below:

Symposium on Evaluation of Metallic Materials in Design for Low-Temperature Service,
Symposium on Elevated-Temperature Compression Testing of Sheet Materials,
Symposium on Major Effects of Minor Constituents on the Properties of Materials,
Symposium on Soil Dynamics,
Symposium on Radiation Effects in Refractory Fuel Compounds,
Symposium on Erosion and Cavitation,
Symposium on Impurities in Steam,
Symposium on Extension of Sensitivity for Determining Various Constituents in Metals, and
Symposium on Microviscometry.

RECORD OF PUBLICATIONS ISSUED April 15, 1960 to April 15, 1961

The extent of the Society's publications during the year is presented in the accompanying Record of Publications.

Regular Publications:

1960 Proceedings, 1272 pp., 7650 copies.
1960 Gillett Lecture, 20 pp., 2500 copies.
1960 Marburg Lecture, 20 pp., 2500 copies.
1960 Supplements to Book of Standards:
    Part 1, 462 pp., 13,750 copies.
    Part 2, 364 pp., 12,000 copies.
    Part 3, 192 pp., 9200 copies.
    Part 4, 256 pp., 9400 copies.
    Part 5, 252 pp., 8000 copies.
    Part 6, 224 pp., 7500 copies.
    Part 7, 336 pp., 7000 copies.
    Part 8, 224 pp., 7500 copies.
    Part 9, 512 pp., 10,000 copies.
    Part 10, 350 pp., 7000 copies.
    Methods of Chemical Analysis of Metals, 776 pp., 6000 copies.
1960 Year Book, 792 pp., 8500 copies.
1960 Index to Standards, 232 pp., 24,000 copies.
ASTM BULLETIN, 9 issues (May 1960 to April 1961) total number of pages 856, average number of copies 18,072 (265 pages of technical papers).

A statement of the volume of the principal publications for the past four years is given below:

	1957–1958, pages	1958–1959, pages	1959–1960, pages	1960–1961, pages
Proceedings	1440	1430	1423	1252
Marburg Lecture	40	24	32	20
Gillett Lecture	36	22	26	20
Book of ASTM Standards	....	14 390	....	....
Supplements to Book of Standards	2644	....	2962	3172
ASTM Methods of Chemical Analysis of Metals	....	....	....	776
Index to ASTM Standards	251	240	224	232
Year Book	756	784	672	792
*Special Technical Publications	3557	3693	3413	5060
*Other Special Publications	88	40	12	34
Materials Research & Standards (formerly ASTM BULLETIN)	840	844	836	856
	9652	21 467	9600	12 214

* Not included under this listing are a number of publications which are partly of the nature of compilations of standards such as:

- Building Code Compilation,
- Manual of ASTM Standards on Refractory Materials,
- Procedures for Testing Soils,
- Manual on Industrial Water (STP 148),
- Selected ASTM Engineering Materials Standards for Use in College Curricula,
- Manual on Measurements and Sampling of Petroleum and Petroleum Products, and
- Knock Test Manuals.

Special Compilations of Standards Published from April 15, 1960, to April 15, 1961:

Specifications for Steel Piping Materials (A-1), 536 pp., 2600 copies.

ASTM Standards on Cement (C-1), 296 pp., 2800 copies.

ASTM Standards on Mineral Aggregates, Concrete, and Nonbituminous Highway Materials (C-9, D-4), 408 pp., 3000 copies.

ASTM Standards on Gypsum (C-11), 160 pp., 2000 copies.

ASTM Standards on Petroleum Products and Lubricants (D-2):
    Vol. I, 1152 pp., 7000 copies.
    Vol. II, 712 pp., 2500 copies.

ASTM Standards on Bituminous Materials for Highway Constructions, Waterproofing and Roofing (D-4, D-8), 488 pp., 2400 copies.

ASTM Standards on Soaps and Other Detergents (D-12), 280 pp., 1200 copies.

ASTM Standards on Textile Materials (D-13), 968 pp., 2100 copies.

Manual on Industrial Water and Industrial Waste Water (D-19) (STP 148-E), 670 pp., 2000 copies.

Selected ASTM Engineering Materials Standards for Use in College Curricula, 452 pp., 15,000 copies.

ASTM Manual for Rating Motor Fuels by Motor and Research Methods, 224 pp., 2600 copies.

Building Code Supplement, 270 pp., 1800 copies.

Special Compilations in Prospect:

ASTM Standards on Copper and Copper Alloys (B-5), 750 pp., 1600 copies.

ASTM Standards on Plastics (D-20), 1240 pp., 2500 copies.

Method for Emission Spectrochemical Analysis (E-2), 704 pp., 3000 copies.

Special Publications Issued from April 15, 1960, to April 15, 1961:

Punch Card Tabulation of Elevated Temperature Strength Data of Metals and Alloys, 72 cards (Keysort), 200 sets.

Seventh Supplement to Numerical Index of Abstracted Infrared Spectra Indexed on Wyandotte ASTM Punched Cards, 34 pp., 200 copies.

X-Ray Powder Data:
    Cards revised—Sets 1 to 5, 4000 cards, 700 sets of 3 by 5 index cards, 500 sets of Keysort cards.
    Book (STP 48-J)—Organic, 384 pp., 2000 copies.
        Inorganic, 696 pp., 2000 copies.

Bibliography of Fatigue References (STP 9-K), 88 pp., 500 copies.

Bibliography and Abstracts on Electrical Contacts (STP 56-N), 67 pp., 1000 copies.

Report on Standard Samples and Related Materials for Spectrochemical Analysis (STP 58-D), 128 pp., 2500 copies.

1960 Supplement to the Metal Cleaning Bibliographical Abstracts (STP 90-E), 48 pp., 2000 copies.

Bibliographical Abstracts of Methods for Analysis of Synthetic Detergents (STP 150-C), 47 pp., 1500 copies.

Compilation of Chemical Composition and Rupture Strengths of Super Strength Alloys (STP 170-B), 8 pp., 1000 copies.

Symposium on High Voltage Cable Insulation (STP 253), 46 pp., 2000 copies.

Soils—1959 Annual Meeting (STP 254), 383 pp., 2000 copies.

Marine Atmosphere Exposure of Galvanic Couples Involving Magnesium, by A. Gallaccio and I. Cornet (STP 255), 28 pp., 1500 copies.

Symposium on Spectroscopic Excitation (STP 259), 68 pp., 2000 copies.

Report on Stress Corrosion Cracking of Austenitic Chromium-Nickel Stainless Steels (STP 264), 100 pp., 2500 copies.

Symposium on Effect of Water-Reducing and Set-Retarding Admixtures on Properties of Concrete (STP 266), 252 pp., 2000 copies.

Symposium on Applied Radiation and Radioisotope Test Methods (STP 268), 118 pp., 2500 copies.

Symposium on Spectroscopy (STP 269), 249 pp., 1500 copies.

Symposium on Durability and Weathering of Structural Sandwich Construction (STP 270), 77 pp., 2000 copies.

Symposium on Adhesion and Adhesives (STP 271), 71 pp., 2000 copies.

Symposium on Newer Metals (STP 272), 224 pp., 2000 copies.

Symposium on Technical Developments in the Handling and Utilization of Water and Industrial Waste Water (STP 273), 96 pp., 2000 copies.

Symposium on Fatigue of Aircraft Structures (STP 274), 144 pp., 2000 copies.

Symposium on Treated Wood for Marine Use (STP 275), 75 pp., 1500 copies.

Materials in Nuclear Application (STP 276) (includes papers from Ceramics in Nuclear Energy, Postirradiation Effects in Polymers, and Radiation Effects and Dosimetry), 352 pp., 3000 copies.

Symposium on Road and Paving Materials (STP 277), 132 pp., 2000 copies.

Symposium on Nondestructive Testing in the Missile Industry (STP 278), 77 pp., 2000 copies.

Symposium on Bituminous Waterproofing and Roofing Materials (STP 280), 87 pp., 2000 copies.

Symposium on Air Pollution Control (STP 281), 48 pp., 3000 copies.

Building Construction Materials (STP 282) (includes Symposium on Wood in Building Construction, and Symposium on Methods for Testing Building Construction), 123 pp., 2000 copies.

Properties of Crystalline Solids (STP 283) (includes Symposium on Recent Progress in Materials Sciences and Symposium on Nature and Origin of Strength of Materials), 150 pp., 3000 copies.

Symposium on Radiation Effects and Radiation Dosimetry (STP 286), 162 pp., 2500 copies.

Elevated Temperature Properties of Aluminum and Magnesium Alloys (STP 291), 312 pp., 1500 copies.

Index to the Literature on X-Ray Spectrographic Analysis, Part I (STP 292), 46 pp., 2000 copies.

Final Report—Strength and Related Properties of Wood Poles, by L. W. Wood, E. C. Erickson, and A. W. Dohr (STP 295), 182 pp., 2000 copies.

Special Publications in Prospect:

X-Ray Powder Data Cards, Set 11, 1080 cards, 800 sets of 3 by 5 index cards, 600 sets of Keysort cards.

Index to X-Ray Powder Data File (STP 48-K), 800 pp., 1500 copies.

Reinforced Plastics for High-Speed Flight Vehicles (STP 279) (includes Symposium on Thermal Ablation and Symposium on Reinforced Plastics), 144 pp., 2000 copies.

Symposium on Acoustical Fatigue (STP 284), 72 pp., 2000 copies.

Symposium on Methods of Metallographic Specimen Preparation (STP 285), 144 pp., 2000 copies.

Symposium on Low-Temperature Properties of High-Strength Aircraft and Missile Materials (STP 287), 232 pp., 2500 copies.

Symposium on Shear and Torsion Testing (STP 289), 128 pp., 2000 copies.

Twenty Year Atmospheric Corrosion of Zinc Coated Wire and Wire Products (STP 290), 200 pp., 2500 copies.

Symposium on Nuclear Methods for Measuring Soil Density and Moisture (STP 293), 112 pp., 1500 copies.

Road and Paving Materials (Bituminous—1960) (STP 294), 98 pp., 1500 copies.

Physical Properties of Metals and Alloys at Cryogenic and Elevated Temperatures (STP 296), 212 pp., 1500 copies.

A Five-Attribute System of Describing Visual Appearance, by Deane B. Judd (STP 297), 20 pp., 2500 copies.

Symposium on Current Research on Motor Gasoline Which May Affect Future Specifications (STP 298), 128 pp., 3000 copies.

Symposium on Non-Newtonian Viscometry (STP 299), 128 pp., 2000 copies.

Symposium on Materials and Electron Device Processing (STP 300), 400 pp., 2000 copies.

Papers on Fire Test Methods (STP 301), 48 pp., 2000 copies.

Symposium on Methods of Testing Building Construction (STP—not assigned), 64 pp., 2000 copies.

Respectfully submitted on behalf of the committee,

R. E. HESS,
Chairman.

APPENDIX V

REPORT OF ADMINISTRATIVE COMMITTEE ON DISTRICT ACTIVITIES

The Administrative Committee on District Activities held four meetings during the year. In addition, two special meetings with representatives of the Chicago and Ohio Valley Districts were held to discuss specific problems in these districts.

At the meeting in June, 1960, Messrs. J. H. Calbeck, W. H. Lutz, and W. C. Magruder retired and were replaced by Messrs. G. H. Harnden, C. E. Proudley, and A. M. Siegel. Mr. M. N. Clair was reappointed for a three-year term.

The committee sponsored its annual breakfast on June 29, 1960, for District Council Officers. While the meeting proved a valuable opportunity to exchange ideas, the committee has decided to hold an open session at the 1961 Annual Meeting to allow for discussion in depth of district problems.

In an effort to bring as many members of ASTM as possible into districts, several major steps were taken by the committee. The Northwest District, composed of Idaho, Oregon, Washington, and British Columbia, has been organized and has held its inaugural meeting in Seattle, Wash., July, 1961. The state of Montana is being added to this district.

The state of Nevada was incorporated into the Northern and Southern California Districts. The committee has also recommended to the Board of Directors two new Districts: the Central Plains District composed of Kansas, Nebraska, Iowa, and western Missouri, and the Northern Plains District composed of North and South Dakota, Minnesota, and western Wisconsin. Thus all of the continental United States except Alaska will be served by the district structure. The possibility of the formation of a Mexican District is also under consideration.

Another accomplishment of the committee was the complete revision of the District Charter and Manual for District Operation. This was the first over-all revision since 1947. The revised Charter and Manual will be presented at an open session of all District Officers and Councilors to be held at the Annual Meeting. It is expected that the Charter and Manual will be presented for approval to the Board of Directors at its September meeting.

During the year, the committee brought to the attention of the districts new ideas to stimulate district activities, and also asked for suggestions from the various councils. As a result, a portable exhibit designed expressly for use at district meetings has been recommended to the Board.

The Student Award Program is now functioning in 12 districts with over 600 awards presented. In addition, the Northwest District is investigating a program and the Washington District held a special Student Program as a regular meeting of the district.

In connection with the National Meetings, the Ohio Valley District was host to Committee Week held February, 1961, in Cincinnati, Ohio. Mr. Bernard Berger, of the Taft Sanitation Laboratories, was the featured speaker at a Dinner Meeting held in connection with Committee Week.

The Philadelphia District, in addition to being the host to the Annual Meeting to be held June 25–30, 1961, at Atlantic City, N. J., was co-sponsor with Committee F-1 on Materials for Electron Tubes and Semiconductor Devices of the first National Technical Meeting. This meeting featured a three-day symposium on Materials and Electron Device Processing (to be published as *ASTM STP No. 300*) as well as an address by John W. Campbell, Editor of *Astounding Science Fiction* at a dinner held in conjunction with the symposium. The Philadelphia District made arrangements for the Ladies Program and entertainment for the Annual Dinner held in conjunction with the Annual Meeting.

Respectfully submitted on behalf of the committee,

MILES N. CLAIR,
Chairman.

ALBERT L. BATIK,
Secretary.

APPENDIX VI

REPORT OF ADMINISTRATIVE COMMITTEE ON SIMULATED SERVICE TESTING

The main topics under discussion during the past year by the Administrative Committee on Simulated Service Testing have been symposia on (1) erosion and cavitation tests of material, (2) relation between service, simulated tests, and conventional tests, and (3) evaluation of packaging. It was decided to concentrate on item (1) for 1961 and return to items (2) and (3) in 1962.

A Symposium on Erosion and Cavitation Tests of Materials has been scheduled for the 1961 Annual Meeting. The following papers are planned:[1]

- Accelerated Cavitation Erosion and Sand Erosion, by W. C. Leith and W. S. McIlquham, Dominion Iron Works, Montreal, Canada.
- The Deformation of Solids by the Impact of Liquids at High Speeds, by J. Brunton, Cambridge University, Cambridge, England
- Cavitation in Hydraulic Turbines, by W. J. Rheingans, Allis Chalmers Mfg. Co., York, Pa.
- Liquid Drop Collisions, by Olive Engel, National Bureau of Standards, Washington, D.C.
- Erosion by Liquid Impact, by S. M. DeCorso and R. E. Kothmann, Westinghouse Research Laboratory, Pittsburgh, Pa.
- Erosion by Solid Particles in a Fluid Stream, by Iain Finnie, Shell Development Co., Emeryville, Calif.

The problem of erosion and cavitation damage is of great concern to manufacturers of power equipment and to the military, and consequently the symposium should prove to be timely and of value in clarifying the directions in which ASTM can contribute most effectively in this area in the future.

Respectfully submitted on behalf of the committee,

R. E. PETERSON,
Chairman.

J. W. CAUM,
Secretary.

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[1] Published as *ASTM STP No. 307.*

APPENDIX VII

REPORT OF ADMINISTRATIVE COMMITTEE ON EDUCATION IN MATERIALS

The Administrative Committee on Education in Materials held no regular meetings during the past year owing to scheduling difficulties. Various methods for developing liaison between the ASTM committees and the membership have been considered and discussed with a few committees, but no specific action has resulted.

One suggestion that has been made is to provide a service to interested ASTM members and potential ASTM members by mailing them a one-sheet monthly issue of "News Briefs" covering significant developments in materials and materials science. This could consist of one-sentence or one-paragraph items of current interest directed at educators and research workers. Such a publication would be helpful in bringing timely items to the attention of those who would profit most by them. It would also serve as good advertising to prospective ASTM members. A similar sheet on nuclear engineering education is well received.

Respectfully submitted on behalf of the committee,

Glenn Murphy,
Chairman.

Fred F. Van Atta,
Secretary.

REPORT OF COMMITTEE A-1

ON

STEEL*

Publication by the Society during the past year of the Tentative Specification for Structural Steel (A 36 - 60 T) is of wide significance to the structural steel industry. This is a structural carbon steel with a minimum yield point of 36,000 psi as compared to A 7 steel, the standard structural steel with a minimum yield point of 33,000 psi. After discussions with many consumer organizations, it was decided to issue a separate specification for this steel rather than change the requirements of the A 7 steel.

Open-ended agreements in specifications under the jurisdiction of Committee A-1 have been the subject of a series of meetings of a task group under the chairmanship of J. S. Worth, Bethlehem Steel Co. At the January 25, 1961 meeting of the Advisory Committee, all the A-1 subcommittees were asked to review their specifications and eliminate all open-ended agreements based upon the following principles set forth by Mr. Worth's group:

1. The phrases "when specified" and "unless otherwise specified" are not considered to constitute the basis of an open-ended agreement except when applied to chemical or mechanical property requirements and/or are used in such a manner to downgrade the material.

2. Whenever possible, "supplementary requirements" none of which lowers the base quality of the specification should be listed at the end of a specification, numbered S1, S2, S3, etc., with appropriate reference to these in the main text of the specifications.

Because of the many new steelmaking processes established or being introduced into the U.S.A., Committee A-1 and its subcommittees are being continually asked to include reference to these in existing specifications. A task group under the chairmanship of C. L. Clark, Timken Roller Bearing Co., has been appointed to review this matter so that a fixed procedure can be used to qualify the new processes and appropriate references to them.

Another task group under the chairmanship of W. F. Hodges, General Electric Co., is drafting an extensive revision of the Committee A-1 Bylaws. The revised Bylaws will reflect the present structure of the committee and expand those sections dealing with the procedure for the promulgation of standards.

At the June, 1960, meeting announcement was made that the U.S.A. has become a participating member of Technical Committee 17 on Steel of the International Organization for Standardization. The A-1 Advisory Committee had approved, previous to the meeting, the formation of a nine man group to advise the American Standards Association (U.S.A. member of ISO) in this activity. The nine man group consists of representatives of four steel producers and four steel consumers with a wide range of interest and a secretary who is a member of the ASTM staff. C. L. Kent, Jones & Laughlin Steel Corp., has been appointed as chairman. Reports of the activity of this group will be given at meetings of the A-1 Advisory Committee and the appropriate A-1 subcommittees will be consulted on subjects within their scope of activity. During the past year, appropriate personnel of Subcommittees XIII and XV have been engaged in such work. Also personnel of Division S of ASTM Committee E-3 on Chemical Analysis of Metals and personnel of Committee E-9 on Fatigue have been consulted.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

A proposed Tentative Specification for General Requirements for Carbon, Ferritic Alloy, and Austenitic Alloy Steel Tubes has been approved by Subcommittee IX and Committee A-1. This is a document which covers in one place all of the requirements common to 21 specifications for steel tubular products. The 21 individual specifications are being revised in line with the general requirement document and it is expected that the 22 separate proposals will be submitted shortly as a group for action by the Society's Administrative Committee on Standards.

The Tentative Methods and Definitions for the Mechanical Testing of Steel Products (A 370 - 54 T) have been extensively revised by Subcommittee XIII after several years of activity on this project.

In an effort to standardize the testing of wire strand intended for prestressed concrete, Subcommittee XIII has drafted a document which Committee A-1 has asked the Society to make generally available. This Suggested Method of Testing Uncoated Seven-Wire Stress-Relieved Strand for Prestressed Concrete appears as Appendix II to this report.[1] The testing procedures outlined are not sufficiently standardized at this time so that they should be published under an ASTM designation. However they are available to serve as a guide for setting up testing procedures for this product.

For many years the Classification of Austenite Grain Size in Steels (ASTM Designation: E 19) has been referenced in specifications for steel products as the acceptable standard on grain size. Over the years the metallographers have noted that some of the actual grain sizes depicted in the chart accompanying E 19 are not in accord with the sizes identifying them. Committee E-4 on Metallography has developed the Tentative Methods for Estimating the Average Grain Size of Metals (E 112) with its accompanying Plate IV as a replacement for E 19. Action is being taken this year to refer to Methods E 112 in the specifications promulgated by Committee A-1 in lieu of the reference to E 19. This will clear the way for Committee E-4 to recommend to the Society the withdrawal of E 19.

There were numerous actions on standards taken during the year and referred to the Administrative Committee on Standards. The standards affected are listed in Appendix I[2] to this report.

At the June 29, 1960, meeting of the Advisory Committee, the Special Subcommittee on Bearing Steels became Subcommittee XXVIII on Bearing Steels. This action was taken because of continuing activity in the special group over the past five years.

Meetings.—Committee A-1 met on June 29, 1960, in Atlantic City, N. J., in conjunction with the 1960 Annual Meeting. On June 27 and 28, 25 subcommittees, sections, and task groups met to prepare actions for the next year's activity and to review results of recent letter ballots.

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[1] See p. 126.

[2] See p. 109.

On January 25, 1961, Committee A-1 met in Pittsburgh, Pa. Twenty-five subcommittee, section, and task group meetings were held on January 23 and 24.

Personnel.—The committee consists of 350 voting members, including 153 producers, 145 consumers, and 52 general interest members.

In Subcommittee XXII, L. H. Carr, Edward Valves, Inc., resigned from the chairmanship of the Low-Temperature Advisory Section. T. D. Parker, Climax Molybdenum Co., has been appointed as the new chairman.

E. S. Bower, Republic Steel Corp., has been appointed as secretary of Subcommittee XV on Bar Steels. Subcommittee II on Structural Steel now has a secretary in the person of R. C. Reber, Bethlehem Steel Co. R. C. Griffin, The Babcock and Wilcox Co., has resigned as secretary of Subcommittee XI on Steel for Boilers and Pressure Vessels and C. Floyd, Graver Tank and Manufacturing Co., has been appointed as his replacement.

It is with regret that Committee A-1 records the death of O. U. Cook, one of its honorary members, during the past year. Mr. Cook first became associated with Committee A-1 in 1921 and was a former Director of the Society.

Two former subcommittee officers, Jonathan Jones and H. R. Redington, also died during the year. Jonathan Jones was vice-chairman of Subcommittee II on Structural Steel and for 25 years contributed outstanding work toward the development of ASTM specifications for structural steel. H. R. Redington, former vice-chairman and secretary of Subcommittee IX on Steel Tubing and Pipe, actively participated in the committee's work for about 20 years.

Other well-known members whose death the committee records are H. F. Clemmer (District of Columbia Highway Dept.), H. W. Ladd (Pan American Petroleum Co.), E. C. Smith (Republic Steel Corp.), A. G. Sturrock (Wyckoff Steel Co.), H. C. Swett (Bethlehem Steel Co., Pacific Coast Div.), W. F. Zerbe (Phoenix Steel Corp.), and F. P. Zimmerli (Barnes-Gibson-Raymond Div., Assoc. Spring Corp.).

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

On July 28, 1960, the Administrative Committee on Standards, acting for the Society, accepted the new Tentative Specification for Structural Steel (A 36 - 60 T) on the recommendation of Committee A-1.

On September 29, 1960, the Administrative Committee on Standards accepted the following recommendations of Committee A-1:

Revision of Tentative Specifications for:

Carbon-Steel Castings Suitable for Fusion Welding for High-Temperature Service (A 216-59 T),

Alloy-Steel Castings for Pressure Containing Parts Suitable for High-Temperature Service (A 217-60 T),

Welded Austenitic Stainless Steel Boiler, Superheater, Heat Exchanger, and Condenser Tubes (A 249-60 T),

Carbon and Alloy Steel Forgings for Magnetic Retaining Rings for Turbine Generators (A 288-57 T),

Carbon and Alloy Steel Forgings for Turbine Generator Rotors and Shafts (A 292-59 T),

Carbon and Alloy Steel Forgings for Turbine Rotors and Shafts (A 293-59 T),

Carbon and Alloy Steel Forgings for Turbine Bucket Wheels (A 294-57 T),

Ferritic and Austenitic Steel Castings for High-Temperature Service (A 351-60 T),

Ferritic Steel Castings for Pressure Containing Parts Suitable for Low-Temperature Service (A 352-59 T), and

Alloy-Steel Castings Specially Heat Treated for Pressure Containing Parts Suitable for High-Temperature Service (A 389–59 T).

Revision and Reversion to Tentatives of Standard Specifications for:

Alloy Steel Forgings for Nonmagnetic Coil Retaining Rings for Turbine Generators (A 289–55).

On March 1, 1961, the Administrative Committee on Standards accepted the following recommendations submitted by Committee A-1.

Revision of Tentative Specifications for:

Low Carbon Steel Externally and Internally Threaded Standard Fasteners (A 307–58 T), and

Galvanized Steel Transmission Tower Bolts and Nuts (A 394–55 T).

The new and revised specifications, except for Specification A 307 - 61 T and A 394 - 61 T,[3] appear in the 1960 Supplement to Book of ASTM Standards, Part 1.

RECOMMENDATIONS AFFECTING STANDARDS

The committee is presenting for publication 6 new tentatives and is recommending the revision of 59 tentatives and 13 standards. One tentative is being revised and adopted as standard and six tentatives are being adopted as standard without change. Two standards are being reapproved, having stood for six or more years without change. One standard is being withdrawn.

The recommendations concerning the standards and tentatives are given in detail in Appendix I.[2]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

In view of the many activities in the 17 subcommittees, only the most significant will be covered in the following paragraphs:

Subcommittee VI on Forgings and Billets has developed a new format for its specifications and expects to use this when each specification is next revised. The subcommittee believes that the new format more clearly defines the various requirements appearing in its specifications. The subcommittee last year completed action on a proposed tentative specification for drop forgings but submittal to Committee A-1 has been delayed pending development of a table of dimensional tolerances. Such a table is now in circulation, and the complete specification may be ready for Committee A-1 action in 1961. A proposed specification for eye bolts may also be referred to Committee A-1 this year.

A proposal has been made that Committee A-1 should investigate a specification for castings intended for high-pressure applications at or near ambient temperature by the petroleum industry. Subcommittee VIII has been assigned this task.

In Subcommittee IX on Steel Tubing and Pipe, consideration is being given to the addition of tensile requirements to Specifications A 83, A 178, A 179, A 192, A 214, and A 226 covering boiler, heat exchanger, and condenser tubes. Another task group has been working on uniform requirements for ladle and check analyses, number of tests, and certification and rejection.

In Subcommittee XV on Bar Steels, a task group is considering revisions of the Tentative Method of End Quench Test for Hardenability of Steel (A 255 - 48 T). It is felt that this method should be modernized since it will be used as the basis for U.S.A. participation in the drafting of an ISO recommendation for this type of test.

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[3] Specifications A 307 and A 394 will appear in the 1961 Book of ASTM Standards, Part 1.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

A proposed tentative specification for leaded carbon steel forged flanges and parts for pressure and general service now appears to be generally satisfactory to Subcommittee XXII. This specification has been under development for several years.

In Subcommittee XXVI on Bolting, a revision of Specification A 325 is being drafted so that the material covered will be structural bolts only. The non-structural bolts currently covered by Specification A 325 will be included under a new specification.

Two new specifications for various types of steel chain are being drafted in Subcommittee XXVII. These include a proposed tentative specification for machine chain and coil chain and a proposed tentative specification for weldless chain.

This report has been submitted to letter ballot of the committee, which consists of 350 members; 213 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. J. KANTER,
Chairman.

W. S. SCOTT,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee A-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specification for:

General Requirements for Carbon, Ferritic Alloy and Austenitic Alloy Steel Tubes (A 450 - 61 T).

Revision of Tentative Specifications for:

Seamless Steel Boiler Tubes (A 83 - 60 T),
Seamless Low-Carbon and Carbon-Molybdenum Steel Still Tubes for Refinery Service (A 161 - 60 T),
Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 - 60 T),
Seamless Cold-Drawn Low-Carbon Steel Heat Exchanger and Condenser Tubes (A 179 - 60 T),
Seamless Steel Boiler Tubes for High-Pressure Service (A 192 - 60 T),
Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes (A 199 - 60 T),
Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (A 200 - 60 T),
Seamless Carbon-Molybdenum Alloy Steel Boiler and Superheater Tubes (A 209 - 60 T),
Seamless Medium-Carbon Steel Boiler and Superheater Tubes (A 210 - 60 T),
Seamless Alloy Steel Boiler Superheater, Heat Exchanger and Condenser Tubes (A 213 - 60 T),
Electric-Resistance-Welded Carbon Steel Heat Exchanger and Condenser Tubes (A 214 - 60 T),
Electric-Resistance-Welded Carbon Steel Boiler and Superheater Tubes for High Pressure Service (A 226 - 60 T),
Welded Austenitic Steel Boiler, Superheater, Heat Exchanger, and Condenser Tubes (A 249 - 60 T),
Electric-Resistance-Welded Carbon-Molybdenum Alloy Steel Boiler and Superheater Tubes (A 250 - 60 T),
Quenched and Tempered Steel Bolts and Studs with Suitable Nuts and Plain Hardened Washers (A 325 - 58 T),

Seamless and Welded Steel Tubes for Low-Temperature Service (A 334 – 60 T), and
Seamless and Electric Welded Low Alloy Steel Tubes for Economizers (A 423 – 60 T).

Revision and Reversion to Tentative of Standard Specification for:

Copper Brazed Steel Tubing (A 254 – 58).

These recommendations were accepted by the Standards Committee on July 6, 1961, with the exception of the revision of Specification A 325 which was accepted on August 3, 1961. The new and revised tentative specifications appear in the 1961 Book of ASTM Standards, Part 1.

APPENDIX I

PROPOSED RECOMMENDATIONS AFFECTING STANDARDS ON STEEL

In this Appendix, recommendations are given affecting certain specifications, both standard and tentative, covering various steel products. The specifications under revision appear in their present form in either the 1958 Book of ASTM Standards, Part 1, or the 1959 or 1960 Supplements thereto.

NEW TENTATIVES

The committee recommends for publication as tentative the following method and specifications as appended hereto:[1]

Tentative Method and Specifications for:

Magnetic Particle Inspection of Large Crankshaft Forgings.

Tentative Specifications for:

Centrifugally Cast Austenitic Steel Pipe for High Temperature Service (Jointly with Committee A-10),

Austenitic Centrifugally Cast Cold-Wrought Pipe for High Temperature Service (Jointly with Committee A-10),

High Tensile Strength Carbon-Manganese Steel Plates for Unfired Pressure Vessels,

High-Strength, High-Temperature Bolting Materials with Expansion Coefficients Comparable to Austenitic Steels (Jointly with Committee A-10), and

Steel Conveyor Chain.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Specification for General Requirements for Delivery of Rolled Steel Plates, Shapes, Sheet Piling, and Bars for Structural Use (A 6 – 60 T):

Section 6(h).—Change to read "Tension and bend test specimens for pins and rollers may be taken so that the axis is midway, if practicable, between the center and surface. Tension and bend test specimens for pins and rollers 3 in. and over in diameter shall be taken so that the axis is 1 in. from the surface."

Tentative Specification for Steel for Bridges and Buildings (A 7 – 58 T):

Section 6(c).—Change to read "Check analysis is not required for bar size shapes, for flat bars ½ in. and under in thickness, or for any bars ordered as merchant quality."

Tentative Specification for General Requirements for Hot-Rolled and Cold-Finished Carbon and Alloy Steel Bars (A 29 – 60 T):

Tables I and II.—Add requirements for lead as follows:

Limit, or Maximum of Specified Range, per cent	Permissible Variations Over Maximum Limit or Under Minimum Limit, per cent
0.15 to 0.35, incl...........	0.03

Reference a footnote to the word "Lead" to read "Check analysis tolerance for lead applies both over and under to a specified range of 0.15 to 0.35 per cent."

Table V.—Change to read as shown in the accompanying Table I.

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[1] The new tentatives appear in the 1961 Book of ASTM Standards, Part 1.

Table X.—Change to read as shown in the accompanying Table II.

Table XVII.—Change the title to read "Length Ranges for Random Length Cuttings for Cold-Finished Bars of Carbon Steel and Alloy Steel."

Insert the following tabular material, and make the present paragraph a footnote:

Lengths, ft	Length Range, in.
5 to 20, incl.	24
Over 20 to 30, incl.	36
Over 30 to 40, incl.	48

TABLE I.—PERMISSIBLE VARIATIONS IN THICKNESS AND WIDTH FOR HOT-ROLLED SQUARE EDGE AND ROUND EDGE FLAT BARS OF CARBON STEEL AND ALLOY STEEL.

(Revision of Table V in Specifications A 29 – 60 T)

Specified Width, in.	Permissible Variations in Thickness, for Thickness Given, Over and Under, in.							Permissible Variations in Width, in.	
	0.203 to 0.230, excl	0.230 to 1/4, excl	1/4 to 1/2, incl	Over 1/2 to 1, incl	Over 1 to 2, incl	Over 2 to 3, incl	Over 3	Over	Under
To 1, incl.	0.007	0.007	0.008	0.010	...	...	...	1/64	1/64
Over 1 to 2, incl.	0.007	0.007	0.012	0.015	1/32	...	...	1/32	1/32
Over 2 to 4, incl.	0.008	0.008	0.015	0.020	1/32	3/64	3/64	1/16	1/32
Over 4 to 6, incl.	0.009	0.009	0.015	0.020	1/32	1/16	1/16	3/32	1/16
Over 6 to 8, incl.	[a]	0.015	0.016	0.025	1/32	1/16	[b]	1/8[b]	3/32[b]

[a] Flats over 6 to 8 in. incl, in width, are not available as hot-rolled carbon or alloy steel bars in thickness under 0.230 in.

[b] On flats over 6 to 8 in. incl, in width and over 3 in. in thickness, tolerances shall be negotiated with the manufacturer.

Tentative Specification for Structural Steel (A 36 – 60 T):

Section 1.—Change to read as follows:

1. This specification covers carbon steel shapes, plates, and bars of structural quality not over 4 in. in thickness for use in riveted, bolted, or welded construction of bridges and buildings, and for general structural purposes. When the steel is used for welded construction, it is important that suitable welding procedures be used.

Tentative Specifications for Welded and Seamless Steel Pipe (A 53 – 60 T):

These specifications have been extensively revised in an attempt to define more clearly the several types of products covered. They are appended hereto in their proposed revised form.[2]

Tentative Specifications for Cold-Drawn Steel Wire for Concrete Reinforcement (A 82 – 58 T):

Section 4.—In the table of Paragraph (*a*) change the requirement for the minimum yield point to read "0.8 of the specified minimum tensile strength."

Delete the present Paragraph (*d*) and add new Paragraphs (*d*) and (*e*) to read as follows:

(*d*) The yield strength shall be determined at an extension of 0.005 in. per in. of gage length. The manufacturer is not required to test for yield strength, but is responsible for supplying a product that will meet the stipulated limit when tested in accordance with the provisions of Section 10(*c*).

(*e*) The material shall not exhibit a definite yield point as evidenced by a distinct drop of the beam or halt in the gage of the testing machine prior to reaching ultimate tensile load. The purchaser may, at his option, accept this feature as sufficient evidence of compliance with the specified minimum yield strength to forego conducting the yield strength tests covered by Section 10(*c*).

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[2] The revised specifications appear in the 1961 Book of ASTM Standards, Part 1.

Section 10.—Change to read as follows:

10. (*a*) The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works that concern the manufacture of the material ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications.

(*b*) Except for yield strength, all tests and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified. Such tests shall be so conducted as not to interfere unnecessarily with the operation of the works.

(*c*) If the purchaser considers it desirable to determine compliance with the yield strength requirements in Section 4(*a*), he may have yield strength tests made in a recognized laboratory, or his representative may make the test at the mill if such tests do not interfere unnecessarily with the mill operations.

TABLE II.—PERMISSIBLE VARIATIONS IN LENGTH FOR HOT-ROLLED ROUNDS, SQUARES, HEXAGONS, FLATS, AND BAR SIZE SECTIONS OF CARBON STEEL.

(Revision of Table X in Specifications A 29 – 60 T)

Specified Size of Rounds, Squares, and Hexagons, in.	Specified Size of Flats, in.		Permissible Variations Over Specified Length, in.[a]				
	Thickness	Width	5 to 10 ft, excl	10 to 20 ft, excl	20 to 30 ft, excl	30 to 40 ft, excl	40 to 60 ft, excl
			MILL SHEARING				
To 1, incl.	To 1, incl	To 3, incl	½	¾	1¼	1¾	2¼
Over 1 to 2, incl.	Over 1	To 3, incl	⅝	1	1½	2	2½
Over 1 to 2, incl.	To 1, incl	Over 3 to 6, incl	⅝	1	1½	2	2½
Over 2 to 5, incl.	Over 1	Over 3 to 6, incl	1	1½	1¾	2¼	2¾
Over 5 to 10, incl.	...	...	2	2½	2¾	3	3¼
...	0.230 to 1, incl	Over 6 to 8, incl	¾	1¼	1¾	3½	4
...	Over 1 to 3, incl	Over 6 to 8, incl	1¼	1¾	2	3½	4
Bar Size Sections	...	...	⅝	1	1½	2	2½
			HOT SAWING				
2 to 5, incl.	1 and over	3 and over	[b]	1½	1¾	2¼	2¾
Over 5 to 10, incl.	...	...	[b]	2½	2¾	3	3¼

[a] No permissible variations under.

[b] Smaller sizes and shorter lengths are not hot sawed.

Tentative Specifications for Seamless Steel Boiler Tubes (A 83 – 60 T):

Section 5(*a*).—In the second sentence after the words "chemical analysis" add the phrase "except for spectrochemical analysis."

Tentative Specifications for Forged or Rolled Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 105 – 59 T):

Section 4.—Add a new Paragraph (*e*) to read:

(*e*) Forgings intended for application to pressure vessels may be repair-welded provided the welding is performed in accordance with all the requirements of Part UF of Section VIII of the ASME Boiler and Pressure Vessel Code and the repair welding is approved by the purchasers.

Tentative Specifications for Seamless Carbon-Steel Pipe for High-Temperature Service (A 106 – 60 T):

Section 5(a).—Change the second sentence to read "Samples for chemical analysis, except for spectrochemical analysis, shall be taken in accordance with the Method of Sampling Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron for Determination of Chemical Composition (ASTM Designation: E 59)."

Tentative Specification for Hot-Rolled Carbon Steel Bars (A 107 – 59 T):

Table I.—Under the portion titled "Open Hearth and Electric Furnace Free-Cutting Grades" add a steel of grade designation 12L14 with the following requirements:

Carbon, per cent	0.15 max
Manganese, per cent	0.80 to 1.20
Phosphorus, per cent	0.04 to 0.09
Sulfur, per cent	0.25 to 0.35
Lead, per cent	0.15 to 0.35

Add a footnote referenced to the table title to read "When required, lead is specified as an added element to a standard steel. A range of 0.15 to 0.35 per cent, incl, is commonly specified. Ladle analysis for lead is not determinable since lead is added to the ladle stream while each ingot is poured."

Section 3.—Add to the end of this section, "Leaded steel, when required (see footnote to Table I)."

Tentative Specification for Cold-Finished Carbon Steel Bars and Shafting (A 108 – 58 T):

Table I.—Revise as indicated above for Table I of Specification A 107.

Section 3(b).—Revise as indicated above for Section 3 of Specification A 107.

Tentative Specifications for Black and Hot-Dipped Zinc-Coated (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (A 120 – 57 T):

Section 2(a).—Change the first sentence to read "The steel for both welded and seamless pipe shall be made by one or more of the following processes: open-hearth, electric-furnace, basic oxygen, acid oxygen, acid oxygen steam, or acid-bessemer."

Table I.—Change the required test pressure for butt-welded pipe in the $1\frac{1}{4}$, $1\frac{1}{2}$, 2, $2\frac{1}{2}$, and 3 in. sizes from the present "800 psi" to "1000 psi."

Table III.—Change the required test pressure for butt-welded pipe in the $1\frac{1}{4}$, $1\frac{1}{2}$, 2, $2\frac{1}{2}$, and 3 in. sizes from the present "1100 psi" to "1300 psi."

Table IV.—Change the required test pressure for butt-welded pipe in the $1\frac{1}{4}$, $1\frac{1}{2}$, 2, $2\frac{1}{2}$, and 3 in. sizes from the present "1200 psi" to "1400 psi."

Table VII.—For 2-in. nominal size pipe change the present coupling length of "$3\frac{1}{4}$ in." to "$2\frac{7}{8}$ in."

Tentative Specifications for Electric-Resistance - Welded Steel Pipe (A 135 – 60 T):

Section 5(a).—In the second sentence after the words "chemical analysis" insert the phrase "except for spectrochemical analysis."

Section 8(b).—Change to read "The hydrostatic pressure shall be maintained for not less than 5 sec and, for pipe with wall thicknesses greater than 0.154 in., the pipe shall be jarred near both ends with a 2-lb steel hammer or its equivalent."

New Section.—Add a new Section 9 entitled "Nondestructive Electric Test" to read as follows, renumbering the subsequent sections accordingly:

9. As an alternate to the hydrostatic test as applied to Schedule 10 in $\frac{3}{4}$ to 4-in. nominal pipe size, and when accepted by the purchaser, each pipe shall be tested at the mill by passing it through a nondestructive tester capable of de-

tecting any defect that completely penetrates the pipe wall or any defect equal to or greater than those shown in Table II. Such tests shall be made on the welded seam and the adjacent metal affected thereby, or on the entire cross-section of the pipe, at the option of the manufacturer.

New Table.—Add a new Table II to read as shown in the accompanying Table III, renumbering subsequent tables accordingly.

Section 12.—Renumber as Section 13 and add to Paragraph (*a*) a new sentence to read "The weight of pipe furnished to schedule 10 shall not vary more than plus or minus 10 per cent from that prescribed in Appendix III."

TABLE III.—DIMENSIONS OF MAXIMUM ALLOWABLE DEFECTS IN PIPE WALLS.
(New Table II for Specification A 135).

Wall Thickness[a]		Minor Dimension (Least Dimension of the Defect), Length or Depth	Defective Area (Length × Radial Depth), sq in.
Inches	BWG		
0.035	20	0.006 in.	0.0025
0.049	18	0.006 in.	0.0030
0.065	16	12½ per cent of wall	0.0030
0.083	14	12½ per cent of wall	0.0040
0.095	13	12½ per cent of wall	0.0040
0.109 and over	12 and heavier	12½ per cent of wall	0.0050

[a] For intermediate wall thicknesses, the next lower wall thickness shown in the table shall apply

Section 13.—Renumber as Section 14 and add a new sentence to read "Pipe furnished to schedule 10 shall be in a specified length between 16 and 22 ft with shorts down to 8 ft. Short lengths shall not exceed 10 per cent of the footage."

Section 14.—Renumber as Section 15 and add a new Paragraph (*a*) to read:

> (*a*) *Schedule 10 Pipe.*—Pipe furnished to schedule 10 shall be plain end only. All inside and outside cutting burrs shall be removed. This generally involves breaking the corners.

Section 17.—Renumber as Section 18 and add "Pipe furnished to schedule 10 shall be normally shipped with a light coating of processing oil. If so specified, the pipe can be given a mill coating or a special coating."

New Appendix.—Add a new Appendix II consisting of a new Table IV to read as shown in the accompanying Table IV.

Tentative Specifications for Seamless Low-Carbon and Carbon-Molybdenum Steel Still Tubes for Refinery Service (A 161 – 60 T):

Section 6(a).—In the second sentence after the words "chemical analysis" insert the phrase "except for spectrochemical analysis."

Tentative Specifications for Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 – 60 T):

Section 5(a).—In the second sentence after the words "chemical analysis" insert the phrase "except for spectrochemical analysis."

Section 11(b).—Change to read "Tube with wall thicknesses over 0.154 in. shall be struck near both ends while under the test pressure with a 2-lb steel hammer or its equivalent."

Section 18(b).—Change to read as follows:

> (*b*) Permissible variations in height of flash shall be as follows:
>
> (*1*) For tubes over 2 in. in outside diameter, or over 0.135 in. in wall thickness, the flash on the inside of the tubes shall be cut to a maximum height of 0.010 in. at any point on the tube.

(*2*) For tubes 2 in. and under in outside diameter and with wall thicknesses 0.135 in. and under, the flash on the inside of the tube shall be cut to a maximum height of 0.006 in. at any point on the tube.

Tentative Specifications for Seamless Cold-Drawn Low-Carbon Steel Heat-Exchanger and Condenser Tubes (A 179 – 60 T):

Section 1(*a*).—Change the phrase "to but not including 2 in. in outside diameter" to read "up to 3 in. in outside diameter."

Section 5(*a*).—In the second sentence after the words "chemical analysis" insert the phrase "except for spectrochemical analysis."

Tentative Specifications for Forged or Rolled Steel Pipe Flanges, Forged Fittings, and Valves and Parts for General Service (A 181 – 59 T):

Section 4.—Add a new Paragraph (*f*) to read as indicated above for new Paragraph (*e*) in Section 4 of Specifications A 105.

Tentative Specifications for Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 182 – 59 T):

Section 5(*b*).—Change to read as follows:

> (*b*) All austenitic material shall be furnished in the heat treated condition. The heat treatment, except for the H grades, shall consist of heating the material to a minimum temperature of 1900 F and quenching in water or rapidly cooling by other means.

Table I.—For grade F1 change the required carbon content from the present "0.20 to 0.30" to "0.30 max" per cent, the manganese content from "0.60 to 0.90" to "0.50 to 0.85," the phosphorus and sulfur contents from "0.040 max" to "0.045 max," the silicon content from "0.20 to 0.35" to "0.15 to 0.35," and the molybdenum content from "0.40 to 0.60" to "0.44 to 0.65" per cent.

For grades F5, F5a, F7, F11, and F12 change the required molybdenum content from the present "0.45 to 0.65" to read "0.44 to 0.65" per cent.

For grade F12 change the required chromium content from the present "0.85 to 1.20" to read "0.80 to 1.25" per cent.

For grade F22 change the required molybdenum content from "0.90 to 1.10" to read "0.87 to 1.13" per cent.

TABLE IV.—DIMENSIONS, NOMINAL WEIGHTS AND TEST PRESSURES FOR LIGHT WALL STEEL PIPE.
(New Table IV for Specification A 135 – 60 T)

Nominal Pipe Size, in.	Outside Diameter, in.	Schedule 10			
		Nominal Wall Thickness, in.[a]	Weight, lb per ft	Test Pressure, psi	
				Grade A	Grade B
¾	1.050	0.083	0.86	2500	2500
1	1.315	0.109	1.40	2500	2500
1¼	1.660	0.109	1.81	2400	2500
1½	1.900	0.109	2.09	2100	2400
2	2.375	0.109	2.64	1700	1900
2½	2.875	0.120	3.52	1500	1700
3	3.500	0.120	4.32	1200	1400
3½	4.000	0.120	4.96	1000	1200
4	4.500	0.120	5.61	900	1100

[a] Corresponds to Schedule 10S as listed in the American Standard for Stainless Steel Pipe (ASA B36.19—1957). The test pressures are calculated by the following formula (but shall not exceed 2500 psi):

$$P = \frac{2St}{D}$$

where:

P = pressure in pounds per square inch,
S = fiber stress, 60 per cent of the specified minimum yield point,
t = thickness of wall in inches, and
D = outside diameter in inches.

Tentative Specifications for Welded Steel Wire Fabric for Concrete Reinforcement (A 185 – 58 T):

Section 8(*b*).—Change the word "strand" to read "wire."

Section 18.—Change to read as follows:

18. (*a*) The inspector representing the purchaser shall have free entry at all times while work on the contract of the purchaser is being performed to all parts of the manufacturer's works that concern the manufacture of the material ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications.

TABLE V.—TENSILE REQUIREMENTS.
(Revision of Table II of Specifications A 193)

Grade	Diameter, in.	Minimum Tempering Temperature, deg Fahr	Tensile Strength, min, psi	Yield Point, min, psi	Elongation in 2 in., min, per cent	Reduction of Area, min, per cent
		FERRITIC STEELS				
B5 4 to 6 per cent Chromium. .	Up to 4, incl.	1100	100 000	80 000	16	50
B6 13 per cent Chromium. . . .	Up to 4, incl.	1100	110 000	85 000	15	50
B7 Chromium-Molybdenum .	2½ and under. . . .	1100	125 000	105 000	16	50
	Over 2½ to 4.	1100	115 000	95 000	16	50
	Over 4 to 7.	1100	100 000	75 000	18	50
B14[a] Chromium - Molybdenum - Vanadium.	2½ and under. . . .	1200	125 000	105 000	16	50
B16 Chromium-Molybdenum-Vanadium.	2½ and under. . . .	1200	125 000	105 000	18	50
	Over 2½ to 4.	1200	110 000	95 000	17	45
	Over 4 to 7.	1200	100 000	85 000	16	45

[a] Special normalizing treatment required.

(*b*) Except for yield strength, all tests and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified. Such tests shall be so conducted as not to interfere unnecessarily with the operation of the works.

(*c*) If the purchaser considers it desirable to determine compliance with the yield strength requirements of ASTM Specifications A 82, he may have yield strength tests made in a recognized laboratory, or his representative may make the test at the mill if such tests do not interfere unnecessarily with the mill operations.

Section 19(d).—Change the words "4 per cent" to read "1 per cent" in lines 7 and 9.

Tentative Specifications for Seamless Steel Boiler Tubes for High-Pressure Service (A 192 - 60 T):

Section 5(a).—In the second sentence after the words "chemical analysis" insert the phrase "except for spectrochemical analysis."

Tentative Specifications for Alloy-Steel Bolting Materials for High-Temperature Service (A 193 - 60 T):

Section 1(c).—Delete the words "and washers."

Section 13.—In the title of this section delete the words "and washers." In the text of the section delete the last two sentences covering requirements for washers.

Table II.—Revise the section on Ferritic Steel to read as shown in the accompanying Table V.

Tentative Specifications for Seamless Cold-Drawn Intermediate Alloy-Steel Heat Exchanger and Condenser Tubes (A 199 – 60 T):

Section 1(a).—Change the words "to but not including 2 in. in outside diameter" to read "up to 3 in. in outside diameter."

Section 6(a).—In the second sentence after the words "chemical analysis" insert the phrase "except for spectrochemical analysis."

Tentative Specifications for Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (A 200 – 60 T):

Section 6(a).—In the second sentence after the words "chemical analysis" insert the phrase "except for spectrochemical analysis."

Tentative Specification for Carbon-Silicon Steel Plates of Intermediate Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels (A 201 – 57 T):

Table I.—Change Footnote *a* to read "When steel plates are furnished under this specification to meet the impact requirements of the Specification for Steel Plates for Pressure Vessels for Service at Low Temperatures (ASTM Designation: A 300), the manganese content shall be 0.85 to 1.20 per cent."

Change Footnote *c* to read "When specified, the steel shall be coarse grained having a carburized austenite grain size of 1 to 5 as determined in accordance with the Methods for Estimating the Average Grain Size of Metals (ASTM Designation: E 112) and its Plate IV, by carburizing at 1700 F for 8 hr."

Tentative Specifications for Seamless Carbon - Molybdenum Alloy - Steel Boiler and Superheater Tubes (A 209 – 60 T):

Section 6(a).—In the second sentence after the words "chemical analysis" add the phrase "except for spectrochemical analysis."

Tentative Specifications for Medium-Carbon Seamless Steel Boiler and Superheater Tubes (A 210 – 60 T):

Section 6(a).—In the second sentence after the words "chemical analysis" add the phrase "except for spectrochemical analysis."

Tentative Specification for High Tensile Strength Carbon-Silicon Steel Plates for Boilers and Other Pressure Vessels (A 212 – 57 T):

Table I.—Revise Footnotes *a* and *c* as indicated above for Table I of Specification A 201.

Tentative Specifications for Seamless Alloy Steel Boiler, Superheater, and Heat Exchanger Tubes (A 213 – 60 T):

Section 4(d).—Change the second sentence to read "As evidence that the material has received this treatment it shall exhibit a grain size of No. 7 or coarser as determined in accordance with the Methods for Estimating the Average Grain Size of Metals (ASTM Designation: E 112) and its Plate IV."

Section 7(a).—In the second sentence after the words "chemical analysis" add the phrase "except for spectrochemical analysis."

Tentative Specifications for Electric-Resistance-Welded Steel Heat-Exchanger and Condenser Tubes (A 214 – 60 T):

Section 1(a).—Change the words "up to but not including 2 in. in outside diameter" to read "up to 3 in. in outside diameter."

Section 5(a).—In the second sentence after the words "chemical analysis" add the phrase "except for spectrochemical analysis."

Section 10(b).—Change to read "Tube

with wall thicknesses over 0.154 in. shall be struck near both ends while under the test pressure with a 2-lb steel hammer or its equivalent."

Section 16(b).—Revise as indicated above for Section 18 (*b*) of Specifications A 178.

Tentative Specifications for Electric-Resistance-Welded Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 – 60 T):

Section 5(a).—In the second sentence after the words "chemical analysis" add the phrase "except for spectrochemical analysis."

Section 10(b).—Change to read "Tube with wall thicknesses over 0.154 in. shall be struck near both ends while under the test pressure with a 2-lb steel hammer or its equivalent."

Section 17(b).—Revise as indicated above for Section 18 (*b*) of Specification A 178.

Tentative Specifications for Flat-Rolled Carbon Steel Sheets of Structural Quality (A 245 – 58 T):

These specifications have been extensively revised and are appended hereto in their proposed revised form.[2]

Tentative Specifications for Welded Austenitic Stainless Steel Boiler, Superheater, Heat Exchanger, and Condenser Tubes (A 249 – 60 T):

Section 7(a).—In the second sentence after the words "chemical analysis" add the phrase "except for spectrochemical analysis."

Section 13(b).—Revise to read "Tube with wall thicknesses over 0.154 in. shall be struck near both ends while under the test pressure with a 2-lb steel hammer or its equivalent."

Tentative Specifications for Electric-Resistance-Welded Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 250 – 60 T):

Section 6(a).—In the second sentence after the words "chemical analysis" add the phrase "except for spectrochemical analysis."

Section 12(b).—Change to read "Tube with wall thicknesses over 0.154 in. shall be struck near both ends while under the test pressure with a 2-lb steel hammer or its equivalent."

Section 20(b).—Revise as indicated above for Section 18 (*b*) of Specifications A 178.

Tentative Specification for Alloy Steel Bars to End Quench Hardenability Requirements (A 304 – 57 T):

Table I.—Add a footnote referenced after the table title to read "When required, lead is specified as an added element to a standard steel. A range of 0.15 to 0.35 per cent, incl, is commonly specified. Ladle analysis for lead is not determinable since lead is added to the ladle stream while each ingot is poured."

Section 3(f).—Revise as indicated above for Section 3 of Specification 107.

Tentative Specification for Low-Carbon Steel Externally and Internally Threaded Standard Fasteners (A 307 – 61 T):

Section 4.—Add a final sentence to Paragraph (*a*) to read "The hardness of grade A bolts, except those tested by the wedge tension test, shall not exceed 241 Brinell (100 Rockwell B)."

Revise the last sentence of Paragraph (*d*) to read "But when equipment of sufficient capacity for such tests is not available, they shall meet the following requirements on machined specimen tension tests:"

	Tensile Strength, psi	Elongation in 2 in., per cent
Grade A and Grade B bolts..........	55 000 min	18 min
Grade B bolts only..	90 000 max	...

Tentative Specification for Stress Relief Annealed Cold-Drawn Carbon Steel Bars (A 311 - 60 T):

Table I.—Revise as indicated above for Table I of Specification A 304.

Section 3.—Revise as indicated above for Section 3 of Specification 107.

Tentative Specifications for Seamless and Welded Austenitic Stainless Steel Pipe (A 312 - 60 T) (Jointly with Committee A-10):

Section 8(b).—After the words "chemical analysis" add the phrase "except for spectrochemical analysis."

Tentative Specification for Quenched-and-Tempered Carbon Steel Bars (A 321 - 60 T):

Section 3.—Add to the end of this Section "Leaded steel, when required (Section 8(*b*))."

Section 8.—Letter the present text as Paragraph (*a*) and add a new Paragraph (*b*) to read:

(*b*) When required, lead may be specified as an added element. A range of 0.15 to 0.35 per cent, incl, is commonly specified. Ladle analysis for lead is not determinable since lead is added to the ladle stream while each ingot is poured.

Tentative Specification for Hot-Rolled Alloy Steel Bars (A 322 - 57 T):

Table I.—Revise as indicated above for Table I of Specification A 304.

Section 3(b).—Revise as indicated above for Section 3 of Specification 107.

Tentative Specification for Cold-Finished Alloy Steel Bars (A 331 - 57 T):

Table I.—Revise as indicated above for Table I of Specification A 304.

Section 3(d).—Revise as indicated above for Section 3 of Specification 107.

Tentative Specifications for Seamless and Welded Steel Pipe for Low-Temperature Service (A 333 - 60 T):

Section 8(b).—Change to read as follows:

(*b*) Samples for chemical analysis, except for spectrochemical analysis, shall be taken in accordance with the Method of Sampling Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron for Determination of Chemical Composition (ASTM Designation: E 59). The results of these analyses shall be reported to the purchaser or his representative, and shall conform to the requirements specified in Table II.

Tentative Specifications for Seamless and Welded Steel Tubes for Low-Temperature Service (A 334 - 60 T):

Section 7(a).—In the second sentence after the words "chemical analysis" insert the phrase "except for spectrochemical analysis."

Section 22.—Letter the present text as Paragraph (*a*) and add a new Paragraph (*b*) on height of flash to read as shown above for the revision of Section 18 (*b*) of Specifications A 178.

Tentative Specifications for Seamless Ferritic Alloy Steel Pipe for High-Temperature Service (A 335 - 60 T):

Section 2(d).—Change the first sentence to read "The steel from which the pipe is made shall be a coarse-grained steel having a carburized grain size of 1 to 5 as determined in accordance with the Methods for Estimating the Average Grain Size of Metals (ASTM Desig-

TABLE VI.—PERMISSIBLE VARIATIONS IN WIDTH (SHEETS NOT RESQUARED) (COILS AND CUT LENGTHS, INCLUDING PICKLED SHEETS).[b]

(Revision of Table IV of Specifications A 375 - 58 T)

Specified Width, in.	Permissible Variations over Specified Width, in.[a]
CUT EDGE SHEETS	
To 20, incl.	1/8
Over 20 to 30, incl.	3/16
Over 30 to 50, incl.	1/4
Over 50 to 80, incl.	5/16
Over 80	3/8
MILL EDGE SHEETS	
Over 12 to 14, excl.	7/16
Over 14 to 17, excl.	1/2
Over 17 to 19, excl.	9/16
Over 19 to 21, excl.	5/8
Over 21 to 24, excl.	11/16
Over 24 to 26, excl.	13/16
Over 26 to 28, excl.	15/16
Over 28 to 35, excl.	1 1/8
Over 35 to 50, excl.	1 1/4
Over 50 to 60, excl.	1 1/2
Over 60 to 65, excl.	1 5/8
Over 65 to 70, excl.	1 3/4
Over 70 to 80, excl.	1 7/8
Over 80	2

[a] No permissible variations under.

[b] Out-of-square (cut edge not resquared) variations for sheets of all gages and all sizes is 1/16 in. per 6 in. or fraction thereof, of width.

When sheets are specified to be resquared, the variation for overwidth over length, camber and out-of-square, shall not exceed 1/16 in. for sheets up to and including 48 in. in width and up to and including 120 in. in length; nor 1/8 in. for wider or longer sheets.

nation: E 112) and its Plate IV, by carburizing at 1700 F for 8 hr."

Section 7(b).—Revise to read as indicated above for Section 8(b) of Specifications A 333.

Tentative Specification for Alloy Steel Seamless Drum Forgings (A 336 - 57 T):

This specification has been extensively revised and is appended hereto in its proposed revised form.[2]

Tentative Specifications for Forged or Rolled Carbon and Alloy Steel Flanges, Forged Fittings, and Valves and Parts for Low-Temperature Service (A 350 - 59 T):

These specifications have been extensively revised and are appended hereto in their proposed revised form.[2]

Tentative Specifications for Ferritic and Austenitic Steel Castings for High-Temperature Service (A 351 - 60 T):

Section 1(b).—Change the first sentence to read "One grade of ferritic and nine grades of austenitic steel castings are included in these specifications."

Table I.—Redesignate grade CK20 as grade CK45. Revise the carbon requirement from the present "0.20" to read "0.20 to 0.45" per cent, and the silicon requirement from the present "2.00" to read "1.75" max, per cent.

Add a new grade CF3 with the following requirements:

Carbon, max, per cent	0.03
Manganese, max, per cent	1.50
Silicon, max, per cent	2.00
Sulfur, max, per cent	0.040
Phosphorus, max, per cent	0.040
Chromium, per cent	17.0 to 21.0
Nickel, per cent	8.0 to 12.0

Table II.—Redesignate grade CK20 as grade CK45 and list in a separate column with the following requirements:

Tensile strength, min, psi	65 000
Yield point, min, psi	35 000
Elongation in 2 in., min, per cent	10.0

For grade CF8 change the required minimum yield point from the present "28,000" to read "30,000" psi.

Add a new grade CF3 with the following requirements:

Tensile strength, min, psi	70 000
Yield point, min, psi	30 000
Elongation in 2 in., min psi	35.0

Tentative Specifications for Ferritic Alloy Steel Forged and Bored Pipe for High-Temperature Service (A 369 – 60 T):

Section 9(b).—Change to read as follows:

> (*b*) Samples for chemical analysis, except for spectrochemical analysis, shall be taken in accordance with the Method of Sampling Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron for Determination of Chemical Composition (ASTM Designation: E 59).

Tentative Methods and Definitions for Mechanical Testing of Steel Products (A 370 – 54 T):

These methods have been extensively revised and are printed in a supplement to this report in their proposed revised form.[3]

Tentative Specifications for High Strength Low Alloy Hot-Rolled Steel Sheets and Strip (A 375 – 58 T):

Table IV.—Revised to read as shown in the accompanying Table VI.

Tentative Specifications for Seamless Austenitic Steel Pipe for High-Temperature Central-Station Service (A 376 – 60 T):

Section 8(b).—Revise as indicated above for Section 8(*b*) of Specifications A 333, except that in the last sentence refer to "Table I" in place of "Table II."

Tentative Specification for Chromium-Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 387 – 57 T):

Section 3.—Change Paragraph (*a*) to read "The steels shall be made by either of the following processes: open hearth or electric furnace."

Delete Paragraph (*b*), relettering subsequent paragraphs accordingly.

Reletter Paragraph (*c*) as Paragraph (*b*) and revise the first sentence to read "The steel for grade A of group I shall be a coarse-grained steel having a carburized austenite grain size of 1 to 5 as determined in accordance with the Methods for Estimating the Average Grain Size of Metals (ASTM Designation: E 112) and its Plate IV, by carburizing at 1700 F for 8 hr."

Tentative Specifications for Galvanized Steel Transmission Tower Bolts (A 394 – 61 T):

Section 4(a).—Change to read as follows:

> (*a*) Galvanized bolts shall be used for test purposes. Standard hexagon and square headed bolts shall be tested in full size as specified in Section 7(*b*). At the manufacturer's option, bolts with other than standard hexagon or square head may be tested in full size as specified in Section 7(*b*). Bolts subject to tension tests shall meet the tensile strengths specified in Table I. Bolts not subject to tension tests shall meet the following hardness requirements:

	Minimum	Maximum
Brinell	121	207
Rockwell B	69	95

Tentative Specifications for Factory-Made Wrought Austenitic Steel Welding Fittings (A 403 – 60 T):

Table I.—Add grades 304H, 316H, 321H, and 347H, as shown in the accompanying Table VII.

For grades WP 304L and WP 316L change the maximum carbon requirement from the present "0.030" to read "0.035" per cent.

Section 1.—Add a new Paragraph (*c*) to read as follows:

> (*c*) Optional supplementary requirements are provided for welding fittings where a greater degree of examination is desired. These supplementary requirements call for additional tests to be made at the expense of the purchaser and, when desired, one or more of these may be specified in the order.

Section 3.—Delete Paragraph (*c*).

Section 6.—Change to read as follows:

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[3] The revised methods appear in the 1961 Book of ASTM Standards, Part 1.

6. (*a*) All fittings manufactured under these specifications except the H grades shall receive a carbide solution treatment which shall consist in holding the fittings for a sufficient length of time at a temperature at which the chromium carbides, or other carbides present, will go into solution and then cooling in air or liquid medium at a sufficient rate to prevent reprecipitation.

(*b*) The H grades shall be solution treated at 1800 F, medium, for grades WP304H and WP-316H and at 1925 F, minimum, for grades WP321H, WP347H and WP348H.

(*c*) When specified, grades WP321, WP347 and WP348 shall receive a stabilization treatment which shall consist in holding the fittings at 1500 to 1600 F for not less than 2 hr per in. of thickness and then cooling in the furnace or in air.

(*d*) The heat treatment shall be performed before machining except in instances when reheat-treating is necessary.

(*e*) Any welding involved shall be done prior to these heat treatments.

Supplementary Requirements.—Add the following Supplementary Requirements S1 to S7 at the end of these specifications, including Notes 1 and 2, under the title "Supplementary Requirements for Welding Fittings Requiring Special Consideration." They are also to be preceded by the introductory paragraph below:

These requirements shall not be considered unless specified in the order, in which event, any or all of the supplementary tests specified in Paragraphs S1 to S7 shall be made at the place of manufacture, unless otherwise agreed upon, and at the purchaser's expense. The tests specified shall be witnessed by the purchaser's inspector before shipment of material if so specified in the order.

S1. *Check Analysis*. A check analysis shall be made from each heat of base metal and, if of welded construction, from each lot number of welding material of the fittings offered for delivery.

S2. *Tension Test*. One tension test shall be made on one fitting or representative test piece (Note 1) per lot (Note 2) of fittings. If the fittings are of welded construction, the tension specimen shall include the weld and be prepared so that the weld is at the mid-length location of the specimen.

S3. *Intergranular Corrosion Bend Test*. One intergranular corrosion bend test shall be made on one fitting or representative test piece (Note 1) per lot (Note 2) fittings. If the fittings are of welded construction, the bend specimen shall include the weld and be prepared so that the weld is at the mid-length location of the specimen. Specimens containing a weld shall be bent so that the location of weld is at the point of maximum bend. The method of testing shall be in accordance with the Recommended Practice for Conducting Acidified Copper Sulfate Test for Intergranular Attack in Austenitic Stainless Steel (ASTM Designation: A 393).

Types 304L, 316L, 321, 347, and 348 shall be tested in the sensitized condition. In the case of the other chromium-nickel steels (series 300) the test shall be conducted on specimens representative of the material in the annealed condition.

TABLE VII.—PERMISSIBLE RAW MATERIALS.
(Addition to Table I of Specification A 403 - 60 T)

Grade	Type of Material	Pipe	Plate	Bar	Forging
WP 304H....	304H (18-8), carbon 0.04 to 0.10 per cent	A 312, grade TP 304H	A 240, type 304[h]	A 276, type 304[h]	A 182, grade F 304H
WP 316H....	316H (18-8, Mo), carbon 0.04 to 0.10 per cent	A 312, grade TP 316H	A 240, type 316[h]	A 276, type 316[h]	A 182, grade F 316H
WP 321H....	321H (18-8, Ti), carbon 0.04 to 0.10 per cent	A 312, grade TP 321H	A 240, type 321[h]	A 276, type 321[h]	A 182, grade F 321H
WP 347H....	347H (18-8, Cb + Ta), carbon 0.04 to 0.10 per cent	A 312, grade TP 347H	A 240, type 347[h]	A 276, type 347[h]	A 182, grade F 347H

[h] Except that the carbon content shall be 0.04 to 0.10 per cent.

S4. *Ultrasonic Test.* Each fitting or the raw material from which each fitting is made shall be ultrasonically tested to determine its soundness. The method, where applicable, shall be in accordance with the Recommended Practice for Ultrasonic Testing and Inspection of Heavy Steel Forgings (ASTM Designation: A 388). Acceptance limits shall be a matter of agreement between the manufacturer and the purchaser.

S5. *Photomicrographs.* Photomicrographs at 100 diameters shall be made for information only of the actual base metal structure from one fittings as furnished in each lot (Note 2). The photomicrographs shall be identified as to fittings size, wall thickness, lot (Note 2) identification, and heat.

S6. *Surface Finish.* Machined surfaces shall have a maximum roughness of 250 microinches arithmetical average. All other surfaces shall be suitable for ultrasonic test.

S7. *Liquid Penetrant Test.* All surfaces shall be liquid-penetrant tested. The method shall be in accordance with the Method for Liquid Penetrant Inspection of Forgings.[4] Acceptance limits shall be a matter of agreement between the manufacturer and the purchaser.

NOTE 1.—Where the test specimen for the tension or intergranular corrosion bend test cannot be taken from a fitting due to size limitations, a representative test piece shall be obtained.

The test piece shall be from the same heat and heat treated in the same batch or charge as the fittings it represents and shall have approximately the same amount of working. In addition, test pieces representing fittings manufactured from bars, plate, or forgings shall have a cross section equal to the greatest cross section of the fitting, and test pieces representing fittings manufactured from pipe shall have an outside diameter and wall thickness equal to those of the fitting. The test piece for fittings of welded construction shall be prepared to the same weld procedures and from the same heats of materials as the fittings it represents.

NOTE 2.—A lot shall consist of all fittings of the same type, size, and wall thickness, manufactured from one heat of material (and, if welding is performed using one lot number of electrode or one heat of weld wire, and one lot number of flux) heat treated either in a continuous or batch type furnace controlling within a range of 50 F and equipped with recording pyrometers so that complete records of heat treatment are available.

Tentative Specifications for Seamless Ferritic Alloy-Steel Pipe Specially Heat-Treated for High-Temperature Service (A 405 – 56 T):

Section 7(b).—Revise as indicated above for Section 8(*b*) of Specifications A 333, except in the last sentence refer to "Table I" in place of "Table II."

Tentative Specifications for Factory-Made Wrought Carbon Steel and Alloy Steel Welding Fittings of Seamless or Welded Construction for Low Temperature Service (A 420 – 59 T):

Table I.—Change the designation for the present grade "WPLC" to read "WPLO." Also change the present reference to "A 333, grade C" to read "A 333, grade O."

Tentative Specifications for Butt Welds in Still Tubes for Refinery Service (A 422 – 58 T):

Section 5(g).—Add to the hardness requirement of 241 Brinell, max, the following "(for light wall tubes, a Rockwell B of 100, max)."

Tentative Specifications for Seamless and Electric Welded Low Alloy Steel Tubes for Economizers (A 423 – 60 T):

Section 5.—Identify the present chemical requirements as for grade 1 and add requirements for a new grade 2 as follows:

Carbon, max, per cent	0.15
Manganese, per cent	0.50 to 1.00
Phosphorus, max, per cent	0.04
Sulfur, max, per cent	0.05
Copper, per cent	0.30 to 1.00
Nickel, per cent	0.40 to 1.10
Molybdenum, min, per cent	0.10

Section 7(a).—In the second sentence after the words "chemical analysis" add the phrase "except for spectrochemical analysis."

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[4] This method is being developed by Committee A-1 and is expected to be recommended for publication in 1961.

Tentative Specifications for Centrifugally Cast Ferritic Alloy Steel Pipe for High-Temperature Service (A 426 - 59 T):

Section 7(b).—Revise as indicated above for Section 8(*b*) of Specifications A 333, except in the last sentence refer to "Table I" in place of "Table II."

Tentative Specifications for Austenitic Steel Forged and Bored Pipe for High-Temperature Service (A 430 - 60 T):

Section 9(b).—Revise the first sentence to read "Samples for chemical analysis, except for spectrochemical analysis, shall be taken in accordance with the Method of Sampling Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron for Determination of Chemical Composition (ASTM Designation: E 59)."

Supplementary Requirement S7.—Add a note at the end of this supplementary requirement to read as follows:

> NOTE.—The term "hot ductility tests," as it relates to the material covered in these specifications, involves heating the metal to a temperature approximating those attained in the base metal adjacent to a weld and at rates simulating those occurring during welding, followed by fracturing rapidly at a decreasing temperature, usually in the range of 2200 to 2400 F. The measured reduction of area at the break is indicative of its high temperature ductility.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Method of:

Magnetic Particle Testing and Inspection of Heavy Steel Forgings (**A 275 - 58 T**).

Tentative Specifications for:

Carbon Steel Axles, Non-Heat-Treated, for Railway Use (**A 21 - 57 T**),

Carbon Steel Forgings for Railway Use (**A 236 - 57 T**),

Alloy Steel Forgings for Railway Use (**A 238 - 58 T**),

Untreated Carbon Steel Axles for Export and for General Industrial Use (**A 383 - 57 T**), and

Wrought Alloy Steel Rolls for Cold and Hot Reduction (**A 427 - 58 T**).

ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that the Tentative Specifications for Carbon-Chromium Ball- and Roller-Bearing Steels (A 295 - 46 T) be approved for reference to letter ballot of the Society for adoption as standard in the revised form appended hereto.[2]

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated in the following standards and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Specifications for Steel Screw Spikes (A 66 - 54):

Section 4.—Add a new Paragraph (*c*) to read as follows:

> (*c*) Where the design of the spike is such that full-size testing is impracticable, the tension test may be made on a specimen machined from a finished spike. Dimensions of the test specimen shall conform to the requirements of the Methods and Definitions for Mechanical Testing of Steel Products (ASTM Designation: A 370).

Standard Specification for Structural Steel for Ships (A 131 - 59):

Table I.—Change Footnote *b* to read "Plate steel produced to the requirements of grade C shall be made with fine grain practice, and one McQuaid-Ehn grain size test, according to the Methods for Estimating the Average Grain Size of Metals (ASTM Designation: E 112) and its Plate IV, shall be made on material representing each ladle of each heat."

Table II.—Insert minimum yield point requirements of 32,000 psi for structural steel, and 30,000 psi for rivet steel and steel for cold bending.

Standard Specifications for Electric-Fusion (Arc)-Welded Steel Plate Pipe (Sizes 16 in. and Over) (A 134 – 54):

Section 7(b).—Change the last sentence to read as follows: "Pipe with wall thicknesses over 0.154 in. shall be struck, while under pressure, with a 2-lb hammer, or its equivalent, near the weld at both ends of the pipe (Note 3)."

Standard Specifications for Electric-Fusion (Arc)-Welded Steel Pipe (Sizes 4 in. and Over) (A 139 – 58):

Section 5(a).—In the second sentence after the words "chemical analysis" add the phrase "except for spectrochemical analysis."

Section 10(b).—Change the last sentence to read "Pipe with wall thicknesses over 0.154 in. shall be struck, while under pressure, with a 2-lb hammer, or its equivalent, near the weld at both ends of the pipe."

Standard Specification for Nickel-Steel Plates for Boilers and Other Pressure Vessels (A 203 – 60):

Section 1.—Change the first two sentences to read "This specification covers nickel steel plates in two tensile strength ranges, designated grades A and D, and B and E, intended primarily for fusion welding; for use in boiler shells and other pressure vessels. The maximum thickness of flange quality plates to be specified under this specification shall be 2 in. for all grades; the maximum thickness of firebox quality plates of grades A and B shall be 6 in., and of grades D and E shall be 4 in."

Table I.—For grades A, D, and E change the maximum permissible manganese content as follows:

	Grade A	Grade D	Grade E
Up to 2 in., incl, in thickness	0.70	0.70	0.70
Over 2 to 4 in., incl, in thickness	0.80	0.80	0.80
Over 4 to 6 in., incl, in thickness	0.80	...	...

For grade A change the required nickel content on ladle analysis from the present "2.20 to 2.60" to "2.10 to 2.50" per cent and on check analysis from the present "2.13 to 2.67" to "2.03 to 2.57" per cent.

Standard Specification for Molybdenum-Steel Plates for Boilers and Other Pressure Vessels (A 204 – 57):

Table I.—Change Footnote *a* to read "When specified, the steel shall be coarse-grained having a carburized austenite grain size of 1 to 5 as determined in accordance with the Method for Estimating the Average Grain Size of Metals (ASTM Designation: E 112) and its Plate IV, by carburizing at 1700 F for 8 hr."

Standard Specifications for Welded and Seamless Steel Pipe Piles (A 252 – 59):

Section 8(b).—Delete the word "average" before the words "outside diameter."

Standard Specifications for Copper Brazed Steel Tubing (A 254 – 58):

Section 5(a).—In the second sentence after the words "chemical analysis" insert the phrase "except for spectrochemical analysis."

Standard Specifications for Seamless and Welded Ferritic Stainless Steel Tubing for General Service (A 268 – 60) (Jointly with Committee A-10):

Section 7(a).—Revise as indicated above for Section 5(*a*) of Specifications A 254.

Standard Specifications for Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 – 60) (Jointly with Committee A-10):

Section 7(*a*).—Revise as indicated above for Section 5(*a*) of Specifications A 254.

Standard Specification for Seamless and Welded Austenitic Stainless Steel Sanitary Tubing (A 270 – 60) (Jointly with Committee A-10):

Section 7(*a*).—Revise as indicated above for Section 5(*a*) of Specifications A 254.

Standard Specification for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 – 60) (Jointly with Committee A-10):

Section 7(*a*).—Revise as indicated above for Section 5(*a*) of Specifications A 254.

Standard Specification for Metal-Arc Welded Steel Pipe for High-Pressure Transmission Service (A 381 – 58):

Section 1(*b*).—Change to read "Six classes of pipe, based on minimum yield point requirements, are covered as indicated in Table II."

Table II.—Add two new classes with requirements as follows:

Class	Yield Point, min, psi	Tensile Strength, min, psi	Elongation in 2 in., min, per cent
Y 50	50 000	72 000	21
Y 52	52 000	75 000	20

Fig. 2—In the table of dimensions for the guided bend test jig, list new grades Y 50 and Y 52 as having the same requirements as the present grade Y 48.

WITHDRAWAL OF STANDARD

The committee recommends the withdrawal of the Standard Specifications for Welded and Seamless Open-Hearth Iron Pipe (**A 253 – 60**).

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Standard Specification for Carbon and Alloy Steel Ring and Disk Forgings (**A 243 – 55**), and

Standard Method of Macroetch Testing and Inspection of Steel Forgings (**A 317 – 54**).

APPENDIX II

SUGGESTED METHOD OF TESTING UNCOATED SEVEN-WIRE STRESS-RELIEVED STRAND FOR PRESTRESSED CONCRETE[1, 2]

Scope

1. This method provides procedures for the tension testing of uncoated seven-wire stress-relieved strand for prestressed concrete. This method is intended for use in evaluating the strand for the properties prescribed in the Specifications for Uncoated Seven-Wire Stress-Relieved Strand for Prestressed Concrete (ASTM Designation: A 416).[3]

General Precautions

2. (*a*) Premature failure of the test specimens may result if there is any appreciable notching, cutting, or bending of the specimen by the gripping devices of the testing machine.

(*b*) Errors in testing may result if the seven wires constituting the strand are not loaded uniformly.

(*c*) The mechanical properties of the strand may be materially affected by excessive heating during specimen preparation.

(*d*) These difficulties may be minimized by following the suggested methods of gripping described in Section 4.

Gripping Devices

3. (*a*) The true mechanical properties of the strand are determined by a test in which fracture of the specimen occurs in the free span between the jaws of the testing machine. Therefore, it is desirable to establish a test procedure with suitable apparatus which will consistently produce such results. Due to inherent physical characteristics of individual machines, it is not practical to recommend a universal gripping procedure that is suitable for all testing machines. Therefore, it is necessary to determine which of the methods of gripping described in the following Paragraphs (*b*) to (*h*) is most suitable for the testing equipment available.

(*b*) *Standard V-Grips with Serrated Teeth* (*Note 1*).

(*c*) *Standard V-Grips with Serrated Teeth* (*Note 1*), *Using Cushioning Material.*—In this method, some material is placed between the grips and the specimen to minimize the notching effect of the teeth. Among the materials which have been used are lead foil, aluminum foil, carborundum cloth, brass shims, etc. The type and thickness of material required is dependent on the shape, condition, and coarseness of the teeth.

(*d*) *Standard V-Grips with Serrated Teeth* (*Note 1*), *Using Special Preparation of the Gripped Portions of the Specimen.*—One of the methods used is tinning, in which the gripped portions are cleaned, fluxed, and coated by multiple dips in molten tin or tin alloy held just above the melting point. Another method of preparation is encasing the gripped portions in metal tubing or flexible conduit, using epoxy resin as the bonding agent. The encased portion should be approximately twice the length of lay of the strand.

(*e*) *Special Grips with Smooth, Semi-Cylindrical Grooves* (*Note 2*).—The grooves and the gripped portions of the specimen are coated with an abrasive slurry which

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[1] This proposed method is under the jurisdiction of the ASTM Committee A-1 on Steel, and is the direct responsibility of Subcommittee XIII, on Method of Testing. Comments are solicited.

[2] Published as information only, June, 1961.

[3] 1959 Supplement to Book of ASTM Standards, Part 1.

holds the specimen in the smooth grooves, preventing slippage. The slurry consists of an abrasive such as Grade 3-F aluminum oxide and a carrier such as water or glycerin.

(*f*) *Standard Sockets of the Type Used for Wire Rope.*—The gripped portions of the specimen are anchored in the sockets with zinc. The special procedures for socketing usually employed in the wire rope industry must be followed.

(*g*) *Looped Preformed Wire Splices.*—These devices are available in sizes designed to fit each size of strand to be tested.

(*h*) *Chucking Devices.*—Use of chucking devices of the type generally employed for applying tension to strands in casting beds is not recommended for testing purposes.

NOTE 1.—The number of teeth should be approximately 15 to 30 per in., and the minimum effective gripping length should be approximately 4 in.

NOTE 2.—The radius of curvature of the grooves is approximately the same as the radius of the strand being tested, and is located $\frac{1}{32}$ in. above the flat face of the grip. This prevents the two grips from closing tightly when the specimen is in place.

Specimen Preparation

4. (*a*) Non-uniform loading of the seven wires in the strand may result if slippage of the individual wires of the strand, either the outside wire or the center wire, occur during the tension test. Wire slippage may be minimized by fusing together the cut ends of the specimen. This fusing can be concurrent with torch cutting of the specimens.

(*b*) If the molten-metal temperatures employed during hot-dip tinning or socketing with metallic material are too high, over approximately 700 F, the specimen may be heat affected with a subsequent loss of strength and ductility. Careful temperature controls should be maintained if such methods of specimen preparation are used.

Procedure

5. (*a*) *Yield Strength.*—For determining the yield strength use a Class B-1 extensometer (Note 3) as described in the Method of Verification and Classification of Extensometers (ASTM Designation: E 83).[4] Apply an initial load of 10 per cent of the expected minimum breaking strength to the specimen, then attach the extensometer and adjust it to a reading of 0.001 in. per in. of gage length. Then increase the load until the extensometer indicates an extension of 1 per cent. Record the load for this extension as the yield strength. The extensometer may be removed from the specimen after the yield strength has been determined.

(*b*) *Elongation.* — For determining the elongation use a Class D extensometer (Note 3), as described in Method E 83, having a gage length of not less than 24 in. (Note 4). Apply an initial load of 10 per cent of the required minimum breaking strength to the specimen, then attach the extensometer (Note 3) and adjust it to a zero reading. The extensometer may be removed from the specimen prior to rupture after the specified minimum elongation has been exceeded. It is not necessary to determine the final elongation value.

(*c*) *Breaking Strength.*—Determine the maximum load at which one or more wires of the strand are fractured. Record this load as the breaking strength of the strand.

NOTE 3.—The yield-strength extensometer and the elongation extensometer may be the same instrument or two separate instruments. Two separate instruments are advisable since the more sensitive yield-strength extensometer, which could be damaged when the strand fractures, may be removed following the determination of yield strength. The elongation extensometer may be constructed with less sensitive parts or be constructed in such a way that little damage would result if fracture occurs while the extensometer is attached to the specimen.

NOTE 4.—Specimens that break outside of the extensometer or in the jaws and yet meet the minimum specified values are considered as meeting the mechanical property requirements of the product Specifications A 416,[3] regardless of what procedure of gripping has been used. Specimens that break outside of the extensometer or in the jaws and do not meet the minimum specified values are subject to retest in accordance with Specifications A 416. Specimens that break between the jaws of the extensometer and do not meet the minimum specified values are subject to retest as provided in Section 17 of Specifications A 416.

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[4] 1958 Book of ASTM Standards, Part 3.

REPORT OF COMMITTEE A-2
ON
WROUGHT IRON*

Committee A-2 on Wrought Iron held one meeting during the year: on June 28, 1960, at Atlantic City, N. J.

The committee consists of 26 voting members, of whom 6 are producers, 15 consumers, and 5 general interest members.

The committee wishes to record the loss through death of E. B. Fields of the Atchison, Topeka, & Santa Fe Railway, and the loss by resignation of F. W. Tibbetts, B. W. Bennett, and D. E. Parker. L. H. Scheifle has been added as the representative of The Reading Co., and Earl E. Wagner, as the representative of the Michigan Seamless Tube Co.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1960 Annual Meeting, Committee A-2 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision and Reversion to Tentative of Standard Specifications for:

Wrought Iron Plates (A 42 – 55),
Wrought Iron Rolled or Forged Blooms and Forgings (A 73 – 55),
Staybolt Wrought Iron, Solid (A 84 – 55),
Staybolt Wrought Iron, Hollow-Rolled (A 86 – 55),
Wrought Iron Rivets and Rivet Rounds (A 152 – 39),
Uncoated Wrought Iron Sheets (A 162 – 39),
Zinc-Coated (Galvanized) Wrought Iron Sheets (A 163 – 39),
Single and Double Refined Wrought Iron Bars (A 189 – 42), and
Rolled Wrought Iron Shapes and Bars (A 207 – 39).

Revision and Reversion to Tentative of Standard Definitions of:

Terms Relating to Wrought Iron (A 81 – 33).

These recommendations were accepted by the Standards Committee on September 29, 1960, and the revised specifications and definitions appear in the 1960 Supplement to Book of ASTM Standards, Part 1.

ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that the Tentative Specifications for Electric-Fusion (Arc)-Welded Wrought Iron Plate Pipe (**A 419 – 57 T**)[1] be approved for reference to letter ballot of the Society for adoption as standard with the following revisions.

Section 17(b).—Change to read as follows:

(*b*) *Certification.*—In lieu of inspection by the purchaser's representative, when agreed upon in writing by the purchaser and the supplier a certification shall be made the basis of acceptance of the material. This shall consist of a copy of the manufacturer's test report, or a statement by the supplier, accompanied by a copy of test results, that the material has been sampled, tested and inspected in accordance with the provisions of

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[1] **1958 Book of ASTM Standards, Part 1.**

this specification. Each certification shall be signed by an authorized agent of the manufacturer or the supplier.

The recommendation in this report has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

This report has been submitted to letter ballot of the committee, which consists of 26 members; 23 members returned their ballots, of whom 22 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. S. CRANE,
Chairman.

O. M. TISHLARICH,
Secretary.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee A-2 presented to the Society through the Administrative Committee on Standards revisions of the Tentative Specification for Welded Wrought-Iron Pipe (A 72 – 59 T). The revisions were accepted by the Standards Committee on September 12, 1961, and the revised tentative specification is available as a separate reprint.

REPORT OF COMMITTEE A-3

ON

CAST IRON*

Committee A-3 on Cast Iron held two meetings during the year: in Atlantic City, N. J., on June 30, 1960, and in Cincinnati, Ohio, on Feb. 2, 1961. A number of subcommittees and task groups met in conjunction with the main committee and between meetings of the main committee.

The committee consists of 115 voting members, of whom 38 are classed as producers, 41 as consumers, and 36 as general interest members, with 1 consulting member.

The long-time service of J. S. Vanick was recognized by the Society by an Award of Merit at the 1960 Annual Meeting.

During the year, Subcommittee 19 on Chilled and White Iron Castings, was disbanded because of insufficient interest.

T. E. Eagan has been appointed Chairman of Subcommittee 16 on Heat Treatment of Cast Iron, replacing W. S. Mabb. C. F. Walton was appointed to head a task force on the subject of tolerances and surface finish.

REVISION OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Specifications for Gray Iron Castings for Pressure-Containing Parts for Temperatures Up to 650 F (A 278 – 59 T):[1]

Section 12.—Change to read as follows:

12. Upon request of the purchaser and so specified in the purchase order, a certification shall be made the basis of acceptance of the material. This shall consist of a copy of the manufacturer's test report or a statement by the supplier, accompanied by a copy of the test results, that the material has been sampled, tested, and inspected in accordance with the provisions of these specifications. Each certification so furnished shall be signed by an authorized agent of the supplier or manufacturer.

Tentative Specification for Austenitic Gray Iron Castings (A 436 – 59 T):[1]

Tables I and II.—Add Types 1b and 2b with the following chemical requirements and mechanical requirements, eliminating the present footnotes to the tables.

	Type 1b	Type 2b
Carbon, total, max, per cent	3.00	3.00
Silicon, per cent	1.00 to 2.80	1.00 to 2.80
Manganese, per cent	1.00 to 1.50	0.80 to 1.50
Nickel, per cent	13.50 to 17.50	18.00 to 22.00
Copper, per cent	5.50 to 7.50	0.50 max
Chromium, per cent	2.50 to 3.50	3.00 to 6.00[a]
Sulfur, max, per cent	0.12	0.12
Tensile strength, min, psi	30,000	30,000
Brinell hardness (3000 kg)	149 to 212	171 to 248

Section 5.—Change to read as follows:

5. *Magnetic Properties.*—A convenient shop test for differentiating the various types of austenitic gray iron is based on the fact that a ground face of either the test bars or the castings of types 1, 2 and 4 will not attract a small

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] 1959 Supplement to Book of ASTM Standards, Part 1.

steel horseshoe-type magnet, which is normally attracted to steel (Note). Types 1*b*, 2*b*, 3 and 5 may be attracted to a magnet. This non-magnetic test is a convenient qualitative test only for types 1, 2 and 4 and shall not be used as a basis for acceptance. In the event that non-magnetic castings are specified, the magnetic permeability test should be used.

NOTE.—Alnico magnet should not be used.

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends revisions as follows of the Standard Specifications for Gray Iron Castings for Valves, Flanges, and Pipe Fittings (**A 126 – 42**),[2] and reversion of the specifications to tentative.

Section 5(*c*).—Change to read as follows:

(*c*) In case the transverse test specimen varies from the specified diameter of 1.20 in., a correction factor conforming to the requirements for Test Bar B in Table I of the Method for Transverse Testing of Gray Cast Iron (ASTM Designation: A 438) shall apply.

Section 7(*b*).—Replace the last sentence of this paragraph with the following:

In cases of such substitutions, the requirements for tension bars in Table I of Specifications A 48 shall apply. Equivalent classes under Specifications A 48 for various thicknesses of controlling section are as follows:

Specification A 126 Class	Under 0.50 in.	0.51 to 1.00 in.	Over 1 in.
A...........	25 A	20 B	20 C
B...........	35 A	30 B	30 C
C...........	45 A	40 B	40 C

Explanatory Note.—Replace the list of ASA designation numbers with: "ASA Nos.: B16.1—1960, B16.2—1960, and B16*b*1—1931."

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for:

Cast Iron Culvert Pipe (**A 142 – 59 T**),[1] and

Cast Nodular Iron for Pressure Containing Parts for Use at Elevated Temperatures (**A 395 – 56 T**).[2]

WITHDRAWAL OF STANDARDS

The committee recommends the withdrawal of the following standards due to lack of use:

Standard Specifications for:

Lightweight and Thin-Section Gray Iron Castings (**A 190 – 47**).[2]

Standard Definitions of Terms Relating to:

Cast Iron (**A 196 – 47**).[2]

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Standard Specifications for:

Cast Iron Soil Pipe and Fittings (**A 74 – 42**),

Gray Iron Castings for Elevated Temperatures for Non-Pressure Containing Parts (**A 319 – 53**), and

Nodular Iron Castings (**A 339 – 55**).

Standard Methods of:

Compression Testing of Cast Iron (**A 256 – 46**), and

Impact Testing of Cast Iron (**A 327 – 54**).

Standard Recommended Practices for:

Evaluating the Microstructure of Graphite in Gray Iron (**A 247 – 47**), and

Torsion Tests of Cast Iron (**A 260 – 47**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Task Group on Chemical Analysis of High Carbon Materials (George Krumlauf, chairman).—The joint task force of Committee A-3 and Division S on Sampling of Committee E-3 has under preparation a recommended procedure for sampling cast iron, nodular iron, and pig iron for total carbon.

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[1] 1958 Book of ASTM Standards, Part 1.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Task Group on International Standards (D. E. Krause, chairman) has maintained contact by correspondence with ISO Technical Committee T/C 25. The task group has studied a copy of a Preliminary Draft Recommendation for Charpy Impact Testing of Gray Cast Iron prepared by a committee of the International Committee of Foundry Technical Assns. and has submitted suggestions concerning this proposed International Standard.

Task Group on Thermal Fatigue (W. M. Spear, chairman) is investigating the various types of thermal fatigue problems and is considering the preparation of a symposium on thermal fatigue of cast iron under the sponsorship of Committee A-3.

Subcommittee 6 on General Castings (H. W. Lownie, chairman).—As the result of answers to a questionnaire mailed to some 2200 foundries, Subcommittee 6 has recommended withdrawal of Specification A 190 on the basis of lack of use. A task group has been appointed to investigate reports that the short test bar recommended in Specification A 48 occasionally yields low results in the tension test as compared to the long arbitration bar.

Subcommittee 7 on Microstructure (C. K. Donoho, chairman).—An interim report on a broadened graphite classification system has been prepared as an American Foundrymen's Society paper which should serve as reference pending action by the International Committee of Foundry Technical Organizations.

Subcommittee 12 on Austenitic Cast Iron (Hugo Larson, chairman) has recommended a minor revision of Specification A 436 to eliminate complaints of conflict with other widely used specifications.

Subcommittee 17 on Valves, Flanges, and Fittings (J. J. Kanter, chairman) has recommended revision of Specifications A 126 to bring references to Specifications A 48 up to date.

Subcommittee 18 on Automotive Castings (R. A. Clark, chairman).—A joint task group of Subcommittee 18 and SAE Division 9 has completed a revision of Specification A 159 to bring references to Specification A 48 and A 438 up to date. Other portions of Specification A 159, particularly those dealing with brake drums and clutch plates, are also being revised at this time.

Subcommittee 24 on Nodular Iron (H. W. Ruf, chairman).—A task force headed by J. S. Vanick has been appointed to investigate the subject of nodular iron suitable for pressure vessels such as paper mill dryer rolls, and to submit a draft for a specification.

A Task Force on Nodular Iron Mechanical Properties is preparing a report of a survey concerning satisfaction of producers and consumers with the present grades of nodular iron as covered by ASTM specifications.

A proposal to eliminate the tension test as a mandatory requirement for compliance with Specification A 445 in order to decrease the expense involved in qualifying castings produced by treatment of small ladles of iron is under consideration. Checking of microstructure has been proposed as a qualification test.

This report has been submitted to letter ballot of the committee, which consists of 115 voting members; 74 members returned their ballots, of whom 71 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

RALPH A. CLARK,
Chairman.

C. F. WALTON,
Secretary.

REPORT OF COMMITTEE A-5
ON
CORROSION OF IRON AND STEEL*

Committee A-5 on Corrosion of Iron and Steel held two meetings during the year: on June 30, 1960, at Atlantic City, N. J., and on February 2, 1961, at Cincinnati, Ohio.

The committee consists of 111 members of whom 54 are classified as producers, 31 as consumers, and 26 as general interest members.

NEW TENTATIVES

The committee recommends for publication as tentative the following specifications as appended hereto:[1]

Tentative Specifications for:

Zinc-Coated Flat Steel Armoring Tape, and Copper-Covered Steel Wire Strand.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot vote of the Society:

Standard Specifications for Zinc Coating (Hot Dip) on Iron and Steel Hardware (A 153 – 60),[2] and

Standard Specifications for Zinc Coating (Hot Dip) on Assembled Steel Products (A 386 – 59):[3]

New Sections 3 and 4.—Add the following new Sections 3 and 4 to both specifications, renumbering the subsequent sections accordingly:

3. *Impurities.*—The amount of lead or iron in the bath during actual galvanizing operations shall not exceed the limits permitted for "Prime Western" slab zinc (spelter), namely, lead 1.60 per cent and iron 0.08 per cent. The aluminum content shall not exceed 0.01 per cent.

4. (*a*) *Samples for Chemical Analysis.*—The analyses for impurities in the bath shall be made on samples which are combinations of borings from at least three test ingots taken during actual operations. Samples for test ingots shall be taken on the longitudinal center line of the pot, one at the middle and the other two at opposite ends of the pot, but not nearer the ends than one half the width of the pot. The samples shall be taken from a depth of at least 2 in. below the surface of the zinc, but not greater than 25 per cent of the depth of the zinc.

(*b*) The implements and molds used shall be of materials which will not change the proportions of lead, iron, or aluminum in the test ingot.

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Method of Test for:

Weight of Coating on Zinc-Coated (Galvanized) Iron or Steel Articles (A 90–53),[4]

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] The new tentatives appear in the 1961 Book of ASTM Standards, Part 1.

[2] 1960 Supplement to Book of ASTM Standards, Part 1.

[3] 1959 Supplement to Book of ASTM Standards, Part 1.

[4] 1958 Book of ASTM Standards, Part 3.

Uniformity of Coating by the Preece Test (Copper Sulfate Dip) on Zinc-Coated (Galvanized) Iron or Steel Article (**A 239–41**),[4] and

Weight and Composition of Coating on Long Terne Sheets by the Triple Spot Test (**A 309–54**).[4]

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Method of Test for:

Weight of Coating on Aluminum-Coated Iron or Steel Articles (**A 428–58 T**).[4]

Tentative Specifications for:

Zinc-Coated (Galvanized) Iron or Steel Sheets, Coils and Cut Lengths (**A 93–59 T**),[5]

Long Terne Iron or Steel Sheets, Coils and Cut Lengths (**A 308–59 T**),[5]

1.25 oz. Class Coating (Pot Yield) Zinc-Coated (Galvanized) Iron or Steel Roofing Sheets, (**A 361–59 T**),[5] and

Zinc-Coated Steel Chain-Link Fence Fabric (**A 392–59 T**).[5]

The above tentatives will probably have some revisions made in the near future which make it desirable to retain them in the tentative status.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[6]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee VII on Methods of Testing (O. B. Ellis, chairman) has been evaluating the nitric acid test for porosity of aluminum coatings on steel. The method, as originally specified, required three hours which was too long a period for plant control purposes. Variations in acid concentration and temperature to reduce the time have been studied. The conclusions are: (1) there are uncontrolled variables that affect the final results; (2) test results can be misleading with respect to evaluation of aluminum coatings; (3) the test appears to be too sensitive and too severe for aluminum coatings in the present stage of development; and (4) three hours, as presently specified, is too long for a controlled test.

A task group will start work on methods for determining uniformity of aluminum coatings. Another task force has been appointed to investigate methods of analysis of aluminum coatings on steel.

Subcommittee XI on Sheet Specifications (E. P. Beachum, chairman) has prepared a specification for aluminum-coated sheets which will be sent out for letter ballot of the subcommittee after revision in accordance with comments that have been received. A task force will be appointed to investigate the desirability of writing a culvert specification and to prepare a draft if the decision is favorable.

Subcommittee XII on Wire Specifications (J. F. Occasione, chairman) has completed preparation of Proposed Tentative Specifications for Zinc-Coated Flat Steel Armoring Tape, and proposed Tentative Specifications for Copper-Covered Steel Wire Strand appended to this report. The subcommittee is presently writing specifications for aluminum-coated steel chain link fencing and aluminum-coated steel wire strand, and is working on combining specifications A 122 and A 218 into one specification.

Subcommittee XIII on Hardware Specifications (B. J. Barmack, chairman) has recommended the revision of Specifications A 153 - 60 and A 386 - 59 to include new sections on impurities and samples for chemical analysis as indicated earlier in this report. The task group which is investigating the premature corrosion of galvanized transmission tower steel is continuing its work.

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[5] 1958 Book of ASTM Standards, Part 1.

[6] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee XIV on Sheet Tests (C. P. Larrabee, chairman) has initiated a new atmospheric corrosion test of metallic-coated steel panels by exposing them during September, 1960, at five test sites. The sheets at State College, Pa., which were exposed in 1926 were inspected. Details of the new tests and the results of the inspection of the 1926 sheets at State College are given in the subcommittee report appended to this report.[7]

Subcommittee XV on Wire Tests (F. M. Reinhart, chairman) is accumulating the wire specimens of aluminum-coated steel wires which will be installed at seven tests sites in the United States and at one site in Great Britain for atmospheric corrosion testing. The installation of the specimens will be complated in 1961. A comprehensive report giving the results of twenty years of atmospheric corrosion testing of zinc-coated steel wires as well as some copper-covered, lead-coated, and chromium and chromium-nickel steel wires has been prepared and will be published as STP 290. The report covering the 1957, 1958, 1959, and 1960 inspections is appended to this report.[8]

Subcommittee XVI on Hardware Tests (W. W. Bradley, ex-chairman) has prepared the report on Field Tests of Atmospheric Corrosion of Hardware—1958 Program which is appended to this report.[9]

This report has been submitted to letter ballot of the committee, which consists of 111 members; 65 members returned their ballots, of whom 59 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. F. Hormann,
Chairman.

C. P. Larrabee,
Secretary.

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[7] See p. 136.

[8] See p. 140.

[9] See p. 156.

APPENDIX I

REPORT OF SUBCOMMITTEE XIV ON FIELD TESTS OF ATMOSPHERIC CORROSION OF METALLIC-COATED STEEL PANELS

1960 PROGRAM

The atmospheric corrosion test of metallic-coated steel panels, discussed in previous reports, was exposed at five locations during September 1960. At each site, there are five 28 by 36-in. corrugated panels cut from sheets galvanized in equipment similar to that used for preparation of panels in the 1926 test, nine panels representing all of the six types of continuous galvanizing lines presently in use, one panel coated with pure aluminum, one panel coated with an aluminum-silicon alloy, and one panel coated with terne metal (lead-tin alloy).

The coating weights, shown in Table I, are the averages of the weight of coating on a 2-in.-wide strip sheared before corrugating from each end of the test panel. The eight lighter coated galvanized sheets averaged 1.14 oz per sq ft and six heavier coated sheets averaged 2.28 oz per sq ft. The pure aluminum-coated sheets averaged 1.39 oz per sq ft of coating; the aluminum-silicon, 0.92; and the terne-coated sheet, which was of the type known in trade as "40 lb per double base box (dbb)," had 1.31 oz per sq ft of coating (35 lb per dbb).

The panels were coded by a representative of the American Zinc Inst. and the identity of the manufacturer of each sheet is in the ASTM headquarters files but is unknown to the subcommittee members.

The test locations are:

State College, Pa.	Rural
Newark, N. J.	Industrial
Kure Beach, N. C.	Marine (800 ft from surf)
Brazos River, Tex.	Marine (3900 ft from surf)
Point Reyes, Calif.	Marine (1930 ft from surf)

Because of differences in the direction of the prevailing winds and the distance from sea water, the corrosivities of the three "marine" sites will probably differ to a considerable extent.

The relative corrosivity of the first and third sites toward steel and zinc is given in an appendix to the Report of Committee B-3.[1] The present Newark site is only a few miles from the Kearny, N. J. site, discussed in that report. Independent studies by United States Steel Corp. show that Newark has about the same corrosivity as Kearny. The relative corrosivities during one-year

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 59, p. 183 (1959).

TABLE I.—IDENTIFICATION AND WEIGHTS OF COATING ON SHEETS IN TEST OF METALLIC-COATED STEELS.

Code	Coating Type	Ounces Per Square Foot				
		State College	Newark	Kure Beach	Point Reyes	Brazos River
AP1	Pot[a]	1.05	1.02	1.03	1.02	1.00
EP1	Pot	1.38	1.41	1.39	1.40	1.40
RP1	Pot	1.06	1.06	1.06	1.05	1.03
JP2	Pot[a]	2.43	2.42	2.39	2.37	2.40
PP2	Pot	2.25	2.24	2.21	2.27	2.28
CL1	Line[b]	1.17	1.19	1.16	1.14	1.18
GL1	Line	1.21	1.21	1.21	1.21	1.20
IL1	Line	1.18	1.20	1.20	1.18	1.18
LL1	Line	1.05	1.06	1.01	0.99	1.00
OL1	Line	1.06	1.06	1.06	1.07	1.05
BL2	Line[b]	2.06	1.98	1.86	2.03	2.17
HL2	Line	2.12	2.12	2.12	2.13	2.14
ML2	Line	2.92	3.00	3.09	3.06	3.02
NL2	Line	1.82	1.83	1.88	1.92	1.94
SA1	Al-Si[c]	0.91	0.92	0.93	0.92	0.90
TA1	Al[d]	1.38	1.39	1.38	1.39	1.42
UT4	Terne[e]	1.28	1.31	1.31	1.29	1.34

[a] Individually dipped in conventional galvanizing pots.
[b] Continuous galvanizing line.
[c] Aluminum-silicon coated sheets.
[d] Pure aluminum coated sheets.
[e] Terne-alloy coated sheets.

exposure compared with those of State College, Pa. as unity were:

Location	Zinc Exposed	Steel Exposed
State College, Pa.	1.0	1.0
Kure Beach (800-foot site), N. C.	1.7	1.9
Point Reyes, Calif.	1.8	1.8
Kearny, N. J.	2.6	3.3

From these figures, an estimate of the relative life of galvanized coatings per unit weight can be made for the locations already investigated by Committee B-3. Current Committee B-3 tests include all the test sites used by this subcommittee.

The following organizations participated in the present test:

Supplying Panels	Exposing Panels
Armco Steel Corp.	American Zinc Inst.
Bethlehem Steel Co.	Armco Steel Corp.
Continental Steel Corp.	Bethlehem Steel Co.
Follensbee Steel Corp.	Columbia-Geneva Division, United States Steel Corp.
Granite City Steel Co.	Dow Chemical Co.
Jones & Laughlin Steel Corp.	National Bureau of Standards
Republic Steel Corp.	United States Steel Corp.
United States Steel Corp.	
Weirton Steel Co.	
Wheeling Steel Corp.	

TABLE II.—LIFE OF CORRUGATED GALVANIZED AND BLACK SHEETS, 26 BY 30 IN.

Exposed at State College, Pa., on April 23, 1926.

Sheet[b]	No. 16 Gage, 2.5 oz per sq ft[a]		No. 22 Gage, 2.5 oz per sq ft[a]			No. 22 Gage, 2.0 oz per sq ft[a]			No. 22 Gage, 1.5 oz per sq ft[a]			No. 22 Gage, 1.25 oz per sq ft[a]			No. 22 Gage, 0.75 oz per sq ft[a]			No. 28 Gage, 0.75 oz per sq ft[a]			No. 22 Gage Uncoated
	Years to		Years to			Years to			Years to			Years to			Years to			Years to			Years to Perforation
	1st Rust	100 per cent Rust	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	
R (T)	24.0	*	26.0	*	*	19.0	*	*	17.5	*	*	14.5	*	*	[c]			[c]			26.0
(B)	27.0	*	29.0	*	*	20.0	*	*	16.5	*	*	15.0	*	*	[c]			[c]			
V (T)	27.0	*	21.5	*	*	18.5	*	*	16.0	*	*	15.0	*	*	9.5	23.0	*	[c]			24.0(V)
(or U)(B)	24.0	*	29.0	*	*	25.0	*	*	15.5	*	*	16.5	*	*	10.5	24.0	*	[c]			25.0(U)
N (T)	[c]		20.5	*	*	22.5	*	*	17.5	*	*	12.0	*	*	6.5	20.5	*	11.0	23.0	*	28.0
(B)	[c]		28.0	*	*	22.5	*	*	15.5	*	*	13.5	*	*	9.0	23.0	*	11.5	30.0	[d]	
T (T)	27.0	*	34.4	*	*	21.0	*	*	16.0	*	*	15.0	*	*	9.0	26.0	*	9.0	24.0	*	*
(B)	31.0	*	*	*	*	23.0	*	*	14.5	*	*	13.5	34.4	*	10.5	22.5	*	12.0	25.0	*	
Y (T)	21.5	*	22.5	*	*	19.5	*	*	17.5	*	*	14.5	*	*	10.0	22.5	*	9.5	24.0	*	25.0
(B)	22.5	*	29.0	*	*	19.0	*	*	18.5	*	*	14.5	29.0	*	10.5	30.0	*	13.0	26.0	*	
S (T)	22.0	*	28.0	*	*	25.0	*	*	19.0	*	*	14.5	32.0	*	10.5	21.5	*	11.5	22.0	*	*
(B)	22.5	*	32.0	*	*	28.0	*	*	19.5	*	*	15.5	30.0	*	11.0	21.5	*	11.5	21.5	*	
C (T)	[c]		22.5	*	*	17.5	*	*	16.0	*	*	14.5	*	*	9.5	24.0	*	[c]			*
(B)	[c]		34.4	*	*	21.5	*	*	18.5	*	*	14.5	30.0	*	10.0	24.0	*	[c]			
K (T)	23.0	*	25.0	*	*	22.5	*	*	17.5	*	*	14.5	31.0	*	11.5	24.0	*	10.5	23.0	*	*
(B)	24.0	*	30.0	*	*	28.0	*	*	21.0	*	*	16.5	33.0	*	11.5	22.5	*	11.5	21.0	*	
W (T)	[c]		29.0	*	*	25.0	*	*	16.5	33.0	*	13.5	31.0	*	[c]			[c]			27.0
(B)	[c]		*	*	*	28.0	*	*	15.0	*	*	14.5	30.0	*	[c]			[c]			
Average	24.6	...	...	...	...	22.5	...	...	17.1	...	...	14.6			10.0	23.5		11.1	24.0		

* Data not yet available.

[a] Coating weight is "oz per sq ft" of sheet (both sides).

[b] (T) = Corrugated sample from top of sheet on west side of rack with bottom surface upward.
(B) = Corrugated sample from bottom of sheet on east side of rack with top surface upward.

[c] There were no specimens exposed. [d] Reported as perforation rather than 100 per cent rust in 1956 *Proceedings*.

The method of mounting the individual panels is described below. Three holes, $\frac{3}{8}$ in. in diameter, were punched 3 in. from each horizontal edge as mounted. Four 28 by 36-in. corrugated panels were attached to a frame made of stainless-steel channels, machine screws, and washers, and brass nuts. To prevent any question of corrosion caused by bimetallic contact, all metal pieces were electrically insulated from the test panels by Teflon washers and bushings.

It is planned to inspect the panels at all sites in 1961 (1.0 yr exposure).

The sheets of the 1926 test at State College were inspected during the exposure of the new test. Two new coating failures (T(T) and C(B)) in the 2.5-oz class were found and one sheet (T(B)) in the 1.25-oz class was declared to be 100 per cent rusted. The current status of the 1926 test is shown in Table II.

Respectfully submitted on behalf of the subcommittee,

C. P. LARRABEE,
Chairman.

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[e] There were no specimens exposed. [d] Reported as perforation rather than 100 per cent rust in 1956 *Proceedings*.

APPENDIX II

REPORT OF SUBCOMMITTEE XV ON ATMOSPHERIC EXPOSURE TESTS OF WIRE AND WIRE PRODUCTS

(1957, 1958, 1959, AND 1960 INSPECTIONS)

This report summarizes the results of the atmospheric corrosion tests on wire and wire products through 1960 and presents in detail the data from 1957, 1958, 1959, and 1960 inspections. Reference to earlier reports of Committee A-5 on Corrosion of Iron and Steel which appeared in the Society's *Proceedings* for 1937, 1939, 1941, 1943, 1945, 1947, 1949, 1951, 1953, 1955, and 1957 will be helpful in studies of these data. These earlier reports contain descriptions of the test methods, micrographs, and other characterizations of specimens in the test as well as the data assembled during previous inspections.

The materials undergoing test are mostly bare and zinc-coated steel wires and wire products, but copper-covered and lead-coated steel wires and chromium and chromium-nickel steel wires are also included. The coatings are expressed in weight terms of ounces per square foot of surface. For comparisons of coating thickness, 1 oz per sq ft of surface may be considered as averaging 0.0017 in. thick for zinc coatings, 0.0013 in. thick for copper coatings, and 0.0010 in. thick for lead coatings.[1]

Specimens of wire and wire products have been exposed for about 24 yr at eleven sites. The locations of these sites and their types of atmosphere are as follows:

Test Location	General Type of Atmosphere	Remarks
Pittsburgh, Pa. (abandoned)	Severe industrial	On Brunot Island in the Ohio River about 2 miles west of the city
Bridgeport, Conn.	Industrial	In the city
Sandy Hook, N. J. (abandoned)	Seacoast	About 300 yd from the Atlantic Ocean
Santa Cruz, Calif.	Rural (marine)	About 3 miles from the Pacific Ocean
State College, Pa.	Rural	Central Pennsylvania
Lafayette, Ind.	Rural	Wabash River Valley
Ames, Iowa	Rural	Central Iowa
Manhattan, Kans.	Rural	Kansas River Valley
Ithaca, N. Y.	Rural	Central New York
College Station, Tex.	Rural	About 120 miles inland
Davis, Calif.	Rural	About 80 miles inland

The Pittsburgh (Brunot Island), Pa., and Sandy Hook, N. J., sites were abandoned in 1951 and 1952, respectively.

At each location, more than 900 specimens were exposed. These included short lengths of wire (42 in. long) and wire strand at all locations, farm field fence at nine sites, and barbed wire and chain link fence at eight sites.

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[1] The lead-coated wires in these tests were lead coated over a bonding coat of zinc. See *Proceedings*, Am. Soc. Testing Mats., Vol. 43, p. 87 (1943) for analysis of lead coatings.

The extent of corrosion is being measured by weight losses, by visual examinations of all specimens at the exposure sites, and by tension tests on wire specimens. The Bridgeport, Conn., State College, Pa., Sandy Hook, N. J., and Pittsburgh, Pa., specimens were inspected by a traveling committee. Inspections were made at the other locations by the university personnel in charge of the exposure plots.

Findings from Weight Loss Specimens:

At the start of the program, zinc-coated wire specimens were exposed at Pittsburgh, Pa., Sandy Hook, N. J., and State College, Pa., to determine the rate of loss of coating. Upon completion of these tests, the results were summarized in the 1947 report. The over-all average annual loss of coating in ounces per square foot of surface was 0.369 at Pittsburgh, Pa.; 0.117 at Sandy Hook, N. J.; and 0.060 at State College, Pa.

Findings from Visual Examinations for Corrosion at the Exposure Locations:

The condensed and summarized inspection results of corrosion through 1960 are shown in Table A. In this table is shown the average number of years until first rust and until 100 per cent rust appeared on all specimens of each group where progress of rusting has been sufficient to determine the values at the various locations. For all other groups the average state of corrosion at the last inspection is shown. The detailed inspection records collected in 1957, 1958, 1959, and 1960 are presented in Table I for unfabricated wire, Table II for barbed wire, Table III for farm field fence, Table IV for chain link fence, and Table V for wire strand.

The corrosion of any individual specimen of barbed wire or of farm field fence listed in Tables II and III may be directly compared with the performance of specific unfabricated wire specimens listed in Table I. The Reference Information Table shows the lot numbers of wires used in fabricating all farm field fence and barbed wire samples.

TABLE B.—NUMBER OF SPECIMENS REMOVED FOR TENSILE STRENGTH TESTS.

Exposure Site	Number of Wires Removed, 1957 and 1958					Total Removed to Date
	Bare Steel	Zinc-Coated	Copper-Coated	Lead-Coated	Corrosion-Resistant Steel	
Pittsburgh, Pa.[a]	0	0	0	0	0	771
Sandy Hook, N. J.[a]	0	0	0	0	0	462
Bridgeport, Conn.	16	75	2	4	10	542
State College, Pa.	19	87	2	4	10	307
Ithaca, N. Y.	3	37	2	0	4	202
Lafayette, Ind.	0	35	2	0	0	296
Ames, Iowa	0	9	2	0	10	154
Manhattan, Kan.	0	3	2	0	10	109
College Station, Tex.	0	2	2	0	4	115
Davis, Calif.	0	0	2	0	3	97
Santa Cruz, Calif.	0	3	2	0	3	108
Total	38	251	18	8	54	3163

[a] Tests at these two sites have been terminated except for corrosion-resistant, copper-coated, and lead-coated wires at Pittsburgh, Pa.

At the time of inspection, records are made of the appearance of each specimen. Prior to the time of rusting, the specimens are noted as appearing "metallic" (M), "gray" (G), "yellow" (Y), etc. After rusting has begun, estimates are made of the percentage of area rusted on each specimen. The term "rust" as given in the inspection instruction states: "Rust means real rust of the base metal—ordinarily a rough corroded area."

TABLE A.—REPORT OF GROUP AVERAGES THROUGH 1960—24-YR EXPOSURE.

Abbreviations and symbols used to designate appearance or state of corrosion: M = metallic; G = gray; MG = intermediate between "metallic" and "gray"; Y = yellowed or rust-stained, but not showing actual rough rust of base metal; R = rough rust of base metal, figures preceding R (as 5, 20, 50, 100) are percentages; PHR, PHY = rusted or yellowed in pinholes SR, SY = speckled appearance of rust, or yellowing; Br, Gn, respectively, signify corroded to a brown or green appearance; B = black; D = dark or dirt or soot excluding a better observation; GY = predominantly gray but showing indication of Y. Under each location the single center column designates the state of corrosion for those groups, which as a group, have not reached 1R or 100R;

Zinc Coating Weight Group, oz per sq ft of surface	No. in Group	Pittsburgh, Pa.		Sandy Hook, N. J.$^{(c)}$		Bridgeport, Conn.		State College, Pa.		Lafayette, Ind.		Ithaca, N. Y.		Ames, Iowa		Manhattan, Kansas		College Station, Texas		Santa Cruz, Calif.		Davis, Calif.	
		Average of Observed Years to Initial Rust (IR) and Complete Rust (CR) or State of Corrosion After Approximately 24 Years of Exposure																					
		IR	CR	IR	CR	IR	CR	IR	CR	IR	CR	IR	CR	IR	CR	IR	CR	IR	CR	IR	CR	IR	CR
Unfabricated Wires																							
0.20 to 0.29	10	0.7	1.3	1.9	2.9	2.1	3.0	4.6	6.1	4.4	5.7	5.8	6.8	6.2	7.8	10.0	17.5	7.8	12.9	12.4	17.1	13.2	89R
0.30 to 0.39	7	1.3	1.7	2.7	3.3	2.6	3.5	6.1	7.7	5.2	6.6	6.9	8.0	7.8	10.3	16.0	97R	10.8	17.9	15.4	93R	23R	
0.40 to 0.49	11	1.6	2.3	3.4	4.7	3.4	4.9	7.4	9.4	6.5	8.4	8.2	10.0	10.2	14.3	45R		13.8	72R	19.5	33R	Y	
0.50 to 0.59	8	1.8	2.4	4.5	5.8	4.6	5.8	9.1	11.5	8.1	9.8	9.9	11.4	11.7	16.4	9R		24R		Y		Y	
0.60 to 0.69	11	2.0	2.6	6.0	6.9	5.6	6.8	10.8	13.4	9.3	11.2	11.2	12.8	14.1	18.9	6R		12R		Y		Y	
0.70 to 0.79	8	2.2	2.8	5.6	7.1	5.5	7.3	10.9	14.0	9.6	12.0	11.5	13.8	14.9	99R	Y		7R		Y		Y	
0.80 to 0.89	9	2.5	3.2	6.0	7.5	6.0	8.0	11.5	15.4	9.7	13.5	11.9	15.1	17.8	87R	2R		10R		Y		Y	
0.90 to 1.10	5	2.9	3.7	7.9	9.6	7.2	9.6	14.4	18.9	12.3	16.3	15.0	17.7	49R		GY		3R		GY		GY	
1.20 to 1.50	3	3.6	5.0	10.2	12.3	9.3	12.2	70R		77R		74R		Y		GY		G		G		G	
1.60 to 2.10	10	4.2	5.7	15R		12.0	16.9	2R		10R		10R		G		G		G		G		G	
2.70 to 3.00	3	7.3	8.4	G		21.0	89R	G		G		G		G		G		G		G		G	
Copper Covered	2	Gn		Gn		Gn		Gn		Gn		Gn		Gn		Br		Br		Gn		Br	
Lead Covered	4	D		100R&Y		3R		15R		39PHR		45R		38R		16PHR		16R		28PHR		2R	
12-14% Cr, Cold Drawn	3	D$^{(d)}$		Y,SR		DG		G		G		5R		G,SR		4R		33R		9R		3R	
12-14% Cr, Air Quenched	3	D$^{(d)}$		Y,SR		DG		G		G		MG		G,SR		SR		11R		28SR		28SR	
18% Cr - 8% Ni	4	D$^{(e)}$		MG		G		M		M		M		MG		M		SY		MG		MG	
Wire Strand																							
0.69 to 0.93	4	3.9	7.1	9.1	66R	9.8	15.8	19.3	40R	15.5	61R	16.9	47R	GY		G		G		G		G	
1.00 to 1.20	3	4.9	7.3	10.9	42R	11.7	17.8	22.0	8R	17.0	32R	19.9	24R	GY		G		GY		G		GY	
1.30 to 1.60	3	5.9	10.3	13.6	25R	15.1	20.1	GY		10R		19.6	18R	GY		G		G		GY		GY	
1.70 to 2.50	2	7.3	12.2	G		23.4	55R	G		G		G		G		G		G		G		G	
Lead Covered	1	D$^{(g)}$		100R&Y		5R		100R&Y		55PHR		78R		Y, PHR		18PHR		30PHR		13R		10R	

Chain Link Fence																							
0.29 to 0.52[a]	3	1.5	2.2	1.9	4.8	2.4	5.3	6.0	9.7	5.7	9.3	5.9	11.9	No Specimens Exposed		No Specimens Exposed		No Specimens Exposed		12.8	65R	14.5	42R
1.13 to 1.22[a]	2	3.8	4.9	8.5	10.9	8.9	12.5	15.9	22.3	14.0		17.9								G		GY	
2.09 to 2.92[a]	2	6.5	8.2	5R		20.9	90R	3R		G		1R								G		G	
1.40 to 1.97[b]	10	3.6	6.9	8.9	12.5	9.8	13.9	60R		79R		74R								G		G	
4.21[b]	1	7.5	11.9	GY		19.5	21.4	Y		GY		19.9								G		G	
Copper Covered	1	Gn(f)		Gn		Gn		Gn		Gn		Gn								Br		Br	
Lead Covered	1	35R(f)		100R&Y		5R		100R&Y		40PHR		28R								70PHR		23PHR	
18% Cr - 8% Ni	1	D(f)		G		MG		MG		M		M								MG		MG	
Farm Field Fence																							
0.20 to 0.29	5	No Specimens Exposed		No Specimens Exposed		No Specimens Exposed		5.1	7.5	4.2	7.6	5.0	10.1	6.3	11.4	10.1	97R	6.2	15.8	11.9	20.8	12.9	95R
0.30 to 0.39	9							5.8	8.7	5.1	9.7	5.5	12.4	8.0	11.8	12.7	89R	8.3	96R	12.8	21.7	15.2	76R
0.40 to 0.49	10							7.2	10.5	6.8	11.9	7.1	14.8	11.0	16.1	26R		11.9	42R	13.2	58R	14R	
0.50 to 0.59	3							8.2	10.7	7.3	12.0	7.9	14.9	12.2	16.8	7R		13.1	32R	13.1	27R	Y	
0.60 to 0.69	9							9.8	14.6	8.8	14.1	9.1	16.1	14.6	99R	6R		10R		10R		2R	
0.70 to 0.79	7							12.1	99R	10.7	17.0	12.2	18.6	16.3	82R	MG		3R		GY		G, SR	
0.80 to 0.89	5							14.5	90R	11.8	18.0	13.9	98R	36R		G		3R		Y, SR		GY	
0.90 to 0.99	6							16.9	85R	11.8	19.7	13.6	93R	28R		G		4R		7R		GY	
1.70 to 1.90	3							2R		2R		2R		G		MG		G		G		G	
2.70 to 2.80	2							G		G		G		G		MG		G		G		G	
12-14% Cr, Cold Drawn	1							G		G		10SR		G, SR		10R		20R		15R		30R	
12-14% Cr, Air Quenched	1							G		MG		MG		G		M		10R		5R		5R	
18% Cr - 8% Ni	1							MG		M		M		M		M		M, SY		MG		MG	
Copper Covered	1							Gn		Gn		Gn		Gn		Br		Br		Gn		Br	
Lead Covered	1							100PHR		35PHR		48R		Y, SR		20PHR		15PHR		10R		2R	

a - Coated before weaving.
b - Coated after weaving.
c - Site abandoned after 14.4 years of exposure.
d - Pitted and rough underneath black soot.
e - Few scattered pits underneath black soot.
f - Discontinued after 10 years of exposure.
g - Discontinued after 16 years of exposure.

TABLE I.—REPORT OF 1957, 1958, 1959 AND 1960 INSPECTIONS OF UNFABRICATED WIRE SPECIMENS.

Abbreviations and symbols used to designate appearance or state of corrosion: M = metallic; G = gray; MG = intermediate between M and G; Y = yellowed or rust stained but not showing actual rough rust of base metal; R = rough rust of base metal, figures preceding R (5, 10, 50, 100) are percentages; PHR, PHY = rusted, yellowed in pinholes; SR, SY = speckled appearance of rust or yellowing; Br, Gn respectively, signify corroded to brown or green appearance; GY = predominately G but showing indication of Y.

Coating Weight Group	Average Weight of Zinc Coating oz per sq ft of Surface (a)	Wire Lot No.	Bridgeport, Conn.				Lafayette, Ind.				Ithaca, N. Y				State College, Pa.			
			20.4 yr	21.4 yr	22.4 yr	23.4 yr	21.0 yr	22.0 yr	22.9 yr	24.0 yr	21.0 yr	21.9 yr	22.9 yr	23.9 yr	20.3 yr	21.3 yr	22.3 yr	23.3 yr
	0.24	511																
0.20	0.25	410																
	0.25	315																
to	0.25	115																
	0.27	512																
0.29	0.27	117																
	0.28	411																
Ave	0.28	215																
0.27	0.29	510																
	0.29	217																
	0.30	412																
0.30	0.31	216																
to	0.32	316																
0.39	0.34	520																
Ave	0.35	423																
0.34	0.37	282																
	0.38	422																
	0.40	521																
	0.40	326																
0.40	0.41	125																
to	0.44	522																
0.49	0.45	225																
	0.46	317																
Ave	0.46	226																
0.45	0.48	327																
	0.49	431																
	0.49	228																
	0.49	421																
	0.50	325																
0.50	0.54	127																
to	0.56	430																
0.59	0.56	329																
Ave	0.58	531																
0.56	0.58	432																
	0.59	530																
	0.59	227																
	0.60	238																
	0.61	335																
0.60	0.64	139																
to	0.65	442																
	0.65	239																
0.69	0.66	337																
Ave	0.66	248																
0.65	0.66	236																
	0.67	338																
	0.67	138																
	0.69	542																
	0.72	540																
0.70	0.72	237																
to	0.72	440																
0.79	0.73	336																
	0.74	495*																
Ave	0.74	443																
0.74	0.74	135																
	0.77	441																
	0.80	541																
	0.80	296*																
0.80	0.80	245																
to	0.82	235																
0.80	0.83	543																
	0.83	345																
Ave	0.84	346																
0.83	0.85	149																
	0.87	444																
0.90	0.90	148													100R	100R	100R	100R
to	0.94	247													100R	100R	100R	100R
1.10	0.96	347													100R	100R	100R	100R
Ave	0.97	246													99R	99R	100R	100R
0.96	1.04	348													99R	100R	100R	100R

TABLE I.—*Continued*

Ames, Iowa				College Station, Texas				Santa Cruz, Calif.				Manhattan, Kan.				Davis, Calif.			
20.8 yr	21.8 yr	22.8 yr	23.8 yr	20.9 yr	21.9 yr	22.9 yr	23.9 yr	20.8 yr	21.8 yr	22.9 yr	23.7 yr	20.7 yr	21.7 yr	22.7 yr	23.8 yr	20.8 yr	21.8 yr	22.9 yr	23.8 yr
								100R	100R	100R	100R	100R	100R	100R	100R	96R	96R	100R	100R
								100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R
								100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R
								100R	100R	100R	100R	100R	100R	100R	100R	93R	100R	100R	100R
								100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R
								98R	99R	100R	100R	100R	100R	100R	100R	76R	85R	89R	94R
								100R	100R	100R	100R	100R	100R	100R	100R	33R	75R	79R	70R
								98R	98R	98R	100R	100R	100R	100R	100R	98R	98R	99R	99R
								100R	100R	100R	100R	100R	100R	100R	100R	67R	92R	98R	99R
								96R	96R	100R	100R	100R	100R	100R	100R	5R	14R	24R	30R
								100R	100R	100R	100R	100R	100R	100R	100R	34R	59R	71R	77R
								96R	96R	96R	100R	99R	100R	100R	100R	1R	7R	11R	17R
								96R	96R	96R	100R	100R	100R	100R	100R	13R	29R	35R	39R
								100R	100R	100R	100R	99R	100R	100R	100R	5R	19R	25R	29R
								75R	92R	99R	100R	90R	100R	100R	100R	Y	Y	Y	YSR
								12R	36R	57R	68R	9R	64R	66R	78R	Y	Y	Y	YSR
								21R	46R	66R	80R	31R	90R	90R	100R	Y	Y	Y	YSR
				100R	100R	100R	100R	34R	51R	66R	76R	19R	36R	36R	70R	1R	1R	1R	1R
				26R	63R	78R	96R	3R	4R	7R	9R	1R	15R	15R	19R	Y	Y	Y	YSR
				66R	85R	100R	100R	16R	43R	60R	68R	39R	100R	100R	100R	Y	Y	Y	YSR
				100R	100R	100R	100R	25R	66R	85R	97R	6R	50R	50R	79R	1R	1R	1R	1R
				3R	6R	12R	25R	YSR	YSR	YSR	YSR	Y	Bl	Bl	Y	Y	Y	Y	Y
				43R	94R	100R	100R	1R	6R	13R	21R	2R	50R	50R	60R	Y	Y	Y	Y
				11R	34R	24R	30R	YSR	YSR	YSR	YSR	Y	Y	Y	3R	Y	Y	Y	Y,SR
				5R	12R	38R	64R	YSR	YSR	YSR	YSR	Y	Bl	Bl	1.4R	Y	Y	Y	Y
				24R	48R	98R	100R	YSR	YSR	2R	3R	Y	7R	7R	19R	Y	Y	Y	Y
				16R	28R	54R	74R	YSR	1R	2R	2R	3R	30R	30R	46R	Y	Y	Y	Y
				100R	100R	100R	100R	58R	82R	90R	92R	85R	100R	100R	100R	Y	Y,SR	Y,SR	Y,SR
				10R	19R	47R	71R	YSR	YSR	YSR	1R	1R	8R	8R	27R	Y	Y	Y	Y
				12R	18R	22R	26R	YSR	YSR	YSR	YSR	Y	20R	20R	24R	Y	Y	Y	Y,SR
				5R	10R	17R	23R	YSR	YSR	YSR	YSR	Y	Bl	Bl	Bl	Y	Y	Y	Y
				16R	21R	32R	39R	1R	1R	1R	1R	2R	25R	25R	20R	Y	Y,SR	Y,SR	Y,SR
				3R	6R	11R	17R	1R	1R	1R	1R	Y	GY	GY	Y	Y	Y	Y	Y
				G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G
				G	G	G	G	G	G	G	G	G	G	G	G	1R	1R	1R	1R
				5R	11R	14R	19R	YSR	YSR	YSR	YSR	Y	Y	Y	3R	Y	Y	Y	Y
100R	100R	100R	100R	6R	8R	10R	15R	YSR	YSR	YSR	YSR	Y	10R	10R	7R	Y	Y	Y	Y,SR
100R	100R	100R	100R	5R	7R	11R	13R	YSR	YSR	YSR	YSR	Y	Y	Y	Y	Y	Y	Y	Y
92R	97R	100R	100R	13R	18R	24R	26R	YSR	YSR	YSR	1R	1R	37R	37R	29R	Y	Y	Y	Y,SR
100R	100R	100R	100R	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G
100R	100R	100R	100R	6R	9R	13R	16R	YSR	YSR	YSR	YSR	Y	10R	10R	10R	Y	Y	Y	Y,SR
100R	100R	100R	100R	18R	26R	32R	35R	YSR	YSR	YSR	YSR	1R	12R	12R	12R	Y	Y	Y	Y,SR
100R	100R	100R	100R	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G
99R	100R	100R	100R	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G
100R	100R	100R	100R	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G
100R	100R	100R	100R	9R	13R	17R	25R	YSR	YSR	YSR	YSR	Y	10R	10R	10R	Y	Y	Y	Y
100R	100R	100R	100R	GY	1R	3R	5R	1R	2R	YSR	YSR	Y	Y	Y	Y	Y	Y	Y	Y
100R	100R	100R	100R	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G
83R	90R	96R	99R	GY	2R	2R	4R	GY	YSR	YSR	YSR	GY	Y	Y	Y	GY	GY	GY	Y,SR
100R	100R	100R	100R	3R	6R	10R	15R	YSR	YSR	YSR	YSR	Y	Bl	Bl	Y	Y	Y	Y	Y
100R	100R	100R	100R	6R	13R	16R	20R	YSR	YSR	YSR	YSR	Y	Y	Y	4R	Y	Y	Y	Y
83R	100R	100R	100R	--	--	--	--	--	--	--	--	GY	GY	GY	GY	--	--	--	--
97R	100R	100R	100R	GY	14R	1R	1R	G	G	G	GSR	GY	GY	GY	GY	GY	GY	GY	GY
99R	100R	100R	100R	1R	1R	2R	5R	YSR	YSR	YSR	YSR	Y	Y	Y	Y	GY	GY	GY	Y
100R	100R	100R	100R	G	1R	1R	2R	G	G	G	GSR	GY	GY	GY	GY	G	G	GY	GY
69R	86R	100R	100R	18R	18R	24R	30R	1R	1R	1R	1R	Y	Y	Y	Y	Y	Y	Y	Y
1R	3R	11R	34R	--	--	--	--	--	--	--	--	Y	Y	Y	Y	--	--	--	--
81R	99R	99R	100R	1R	1R	4R	6R	YSR	YSR	YSR	YSR	Y	Y	Y	Y	Y	Y	Y	Y
5R	50R	70R	74R	3R	9R	13R	18R	YSR	YSR	YSR	YSR	Y	Y	Y	Y	Y	Y	Y	Y,SR
73R	90R	98R	99R	1R	2R	3R	3R	G	G	G	GSR	GY	15R	15R	16R	GY	GY	Y,SR	Y,SR
14R	36R	70R	87R	7R	11R	17R	18R	YSR	YSR	YSR	YSR	Y	Y	Y	Y	Y	Y	Y	Y,SR
1R	20R	41R	87R	GY	GY	1R	1R	Y	Y	YSR	YSR	Y	Y	Y	Y	GY	GY	GY	GY
88R	90R	98R	99R	GY	2R	2R	3R	G	G	G	G	GY	GY	Y	Y	G	G	G	G
17R	43R	95R	100R	4R	5R	8R	11R	YSR	YSR	YSR	YSR	Y	Y	Y	Y	Y	Y	Y	Y
1R	20R	46R	93R	GY	1R	1R	1R	Y	YSR	YSR	YSR	GY	GY	GY	GY	GY	GY	GY	GY
4R	4R	9R	20R	7R	9R	13R	14R	YSR	YSR	YSR	YSR	Y	Y	Y	Y	Y	Y,SR	Y,SR	Y,SR
10R	22R	30R	71R	G	G	G	G	G	G	G	G	GY	GY	GY	GY	G	G	G	G
1R	7R	20R	63R	G	G	G	G	G	G	G	G	GY	GY	GY	GY	G	G	G	G
G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G

TABLE I.—*Continued*

Coating Weight Group	Average Weight of Zinc Coating oz per sq ft of Surface (a)	Wire Lot No.	Bridgeport, Conn.				Lafayette, Ind.				Ithaca, N. Y				State College, Pa.			
			20.4 yr	21.4 yr	22.4 yr	23.4 yr	21.0 yr	22.0 yr	22.9 yr	24.0 yr	21.0 yr	21.9 yr	22.9 yr	23.9 yr	20.3 yr	21.3 yr	22.3 yr	23.3 yr
1.20 to 1.50 Ave 1.34	1.20	801					100R	100R	100R	100R	100R	100R	100R	100R	99R	100R	100R	100R
	1.35	800					89R	98R	99R	100R	46R	62R	75R	87R	11R	37R	100R	100R
	1.48	450					6R	9R	20R	30R	6R	8R	21R	36R	2R	10R	10R	10R
1.60 to 2.10 Ave 1.76	1.65	351					6R	7R	8R	10R	4R	5R	11R	21R	1R	6R	6R	6R
	1.67	252					G	G	G	GY	G	G	G	1R	G	G	G	G
	1.68	251					28R	7R	8R	9R	1R	2R	4R	6R	1R	1R	1R	1R
	1.69	551					GY	GY	G	GY	G	G	G	G	G	G	G	G
	1.71	451					G	GY	G	G	G	G	G	1R	G	G	G	G
	1.73	350					G	G	G	G	G	G	G	G	G	G	G	G
	1.76	250					2R	3R	4R	6R	2R	3R	5R	10R	G	G	1R	1R
	1.81	352					5R	6R	7R	15R	GY	1R	1R	2R	GY	GY	GY	GY
	1.86	151					42R	52R	50R	60R	36R	45R	52R	58R	9R	9R	13R	15R
	2.08	552					G	G	G	G	G	G	G	G	G	G	G	G
2.70 to 3.00 Ave 2.86	2.78	298	GY	25R		100R	G	G	G	G	G	G	G	G	G	G	G	G
	2.80	397	Y	73R		100R	G	G	G	G	G	G	G	G	G	G	G	G
	3.00	299	GY	14R		67R	GY	GY	GY	GY	GY	GY	G	1R	G	G	G	G
Copper Covered	7.42	021	GN	GN		GN	GN	Gn	Gn	Gn	GN	Gn	Gn	Gn	GN	Gn	Gn	Gn
	9.24	022	GN	GN		GN	GN	Gn	Gn	Gn	GN	Gn	Gn	Gn	GN	Gn	Gn	Gn
Lead Coated	1.40	040	B	5R		5R	PHR	50PHR	50PHR	55PHR	29R	42R	57R	63R	PHR	20R	22R	25R
	1.48	041	B	B		B	PHR	30PHR	30PHR	30PHR	32R	30R	36R	41R	PHR	10R	11R	13R
	1.68	042	B	5R		5R	PHR	35PHR	40PHR	35PHR	27R	27R	33R	35R	PHR	5R	8R	10R
	1.90	043	B	B		PHR	PHR	38PHR	35PHR	40PHR	14R	19R	35R	41R	PHR	5R	10R	10R
12-14% Cr Cold Drawn		001	D	B		DG	GSR	G,SR	G	G	MG	MG	4R	5R	G	G	G	G
		004	D	DGY		DG	1R	1R	G	G	MG	MG	4R	7R	G	G	G	G
		005	D	B		DG	GSR	G,SR	G	G	MG	MG	2R	4R	G	G	G	G
12-14% Cr Air Quenched		002	DG	DG		DG	G	G	G	G	MG	MG	M	M	G	G	G	G
		006	DG	G		G	G	G	G	G	MG	MG	MG	MG	G	G	G	G
		007	D	DG		DG	GSR	G,SR	G	G	MG	MG	MG	MG	G	G	G	G
18% Cr, 8% Ni		003	G	G		G	M	M	M	M	M	M	M	M	M	M	M	M
		008	G	G		G	M	M	M	MG	M	M	M	M	M	M	M	M
		009	G	G		G	M	M	M	M	M	M	M	M	MG	G	G	G
		015	G	G		G	MG	MG	MG	MG	M	M	M	M	MG	M	M	M

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[a] The zinc coating weights reported in this column are the stripping test determinations made only on samples of unfabricated wires.

* Chromium.

TABLE I.—*Concluded*

Ames, Iowa				College Station, Texas				Santa Cruz, Calif.				Manhattan, Kan.				Davis, Calif.			
20.8 yr	21.8 yr	22.8 yr	23.8 yr	20.9 yr	21.9 yr	22.9 yr	23.9 yr	20.8 yr	21.8 yr	22.9 yr	23.7 yr	20.7 yr	21.7 yr	22.7 yr	23.8 yr	20.8 yr	21.8 yr	22.9 yr	23.8 yr
1R	1R	1R	1R	G	G	G	G	--	--	--	--	GY	GY	GY	GY	--	--	--	--
G	Y	Y	Y	G	G	G	G	--	--	--	--	GY	GY	GY	GY	--	--	--	--
G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G
G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G
G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G
G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G
G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G
G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G
G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G
G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G
G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G
G	GY	G	G	G	G	G	G	G	G	G	G	GY	GY	GY	GY	G	G	G	G
G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G
G	G	G	G	G	G	G	G	G	GY	GY	GY	G	G	G	G	G	G	G	G
G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G
G	G	G	G	G	G	G	G	--	--	--	--	G	G	G	G	--	--	--	--
GN	Gn	Gn	Gn	BR	Br	Br	Br	GN	Gn	Gn	Gn	BR	Bl	Br	BR	BR	BR	BR	Br
GN	Gn	Gn	Gn	BR	Br	Br	Br	GN	Gn	Gn	Gn	BR	Bl	Br	BR	BR	BR	BR	Br
GS	5R	5R	8R	5R	6PHR	6R	8R	18SR	18SR	18SR	18SR	21R	27R	14PHR	15PHR	1R	1R	1R	1R
GM	12R	20R	37R	19R	19PHR	21R	22R	67SR	65SR	65SR	65SR	50R	42R	13PHR	25PHR	4R	4R	4R	4R
GM	16R	29R	47R	18R	16PHR	20R	23R	25SR	28SR	28SR	28SR	22R	24R	10PHR	23PHR	2R	2R	5R	3R
YM	12R	36R	58R	17R	5R	6R	12R	2R	2R	2R	2R	SR	5R	G	G	16SR	17SR	17SR	17SR
GSR	GSR	GSR	GSR	13SR	20R	26R	27R	9R	8R	8R	10R	SR	G, SR	G, SR	3R	3R	3R	3R	3R
GSR	GSR	GSR	GSR	8SR	14SR	19R	23R	6R	6R	6R	8R	SR	G, SR	G, SR	6R	2R	53SR	53SR	53SR
GSR	GSR	GSR	GSR	23SR	35SR	44R	49R	10R	10R	10R	10R	SR	G, SR	G, SR	3R	7R	6R	6R	6R
GSR	GSR	GSR	GSR	6SR	8R	8R	11R	28SR	27SR	27SR	28SR	SR	G, SR	G, SR	3R	23SR	22SR	23SR	23SR
GSR	GSR	GSR	GSR	TrSR	1R	1R	3R	26SR	26SR	26SR	27SR	SR	G, SR	G, SR	SR	16SR	15SR	15SR	15SR
GSR	GSR	GSR	GSR	5SR	6SR	6R	18R	1R	1R	1R	1R	SR	G, SR	G, SR	SR	42SR	45SR	47BR	47SR
MG	MG	MGSR	MGSR	M	M, SY	M, SY	SY	MG	MG	MG	MG	M	M	M	M	MG	MG	MG	MG
MG	MG	MG	MG	M	M	M, SY	SY	MG	MG	MG	MG	M	M	M	M	MG	MG	MG	MG
MG	MG	MG	MG	M	M	M, SY	SY	MG	MG	MG	MG	M	M	M	M	MG	MG	MG	MG
MG	MG	MG	MG	M	M	M, SY	SY	--	--	--	--	M	M	M	M	--	--	--	--

TABLE II.—REPORT OF 1957, 1958, 1959 AND 1960 INSPECTIONS OF BARBED WIRE SPECIMENS.

Abbreviations of symbols used to designate appearance or state of corrosion: M = metallic; B = gray; Y = yellowed, or rust-stained, but not showing actual rough rust of base metal; R = rough rust of base metal figures preceding R (as 5, 20, 50, 100) are percentages; PHR, PHY = rusted or yellowed in pinholes; SR, SY = speckled appearance of rust or yellowing; Br, Gn, respectively, signify corroded to a brown or green appearance; MG = intermediate between M and G; GY = predominantly gray but showing indication of Y; BR = rust on barbs; Bl = Black. All vacant blocks indicate 100R

Wire No.(a)	Average Coating, oz per sq ft of Surface(b)	Lafayette, Ind.				Ithaca, N. Y				State College, Pa.				Ames, Iow	
		years exposure				years exposure				years exposure				years exposure	
		21.0	22.0	22.9	24.0	21.0	21.9	22.9	23.9	20.3	21.3	22.3	23.3	20.8	21.
C411	0.24														
C410	0.27														
C412	0.27														
C420	0.28														
C421	0.37														
C422	0.44														
C430	0.46														
C431	0.53													20R	40R
C432	0.53													100R	100R
C441	0.64													30R	70R
C443	0.70													30R	80R
C442	0.82	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	Y	10R
C446	0.92	87R	91R	95R	98R	58R	80R	90R	95R	60R	100R	100R	100R	Y	Y
C445	0.97	90R	90R	95R	95R	38R	80R	80R	90R	10R	50R	60R	100R	Y	Y
C452	1.52	10R	15R	20R	25R	6R	5R	8R	13R	G	G	G	G	G	G
C450	1.64	87R	91R	90R	95R	88R	95R	95R	95R	75R	80R	90R	95R	Y	Y
C451	1.95	11R	11R	10R	10R	85R	95R	95R	96R	GY	20R	20R	20R	Y	Y
C494	1.99	G	G	G	G	G	G	1R	1R	G	G	G	G	G	G
C494	1.99	G	G	G	G	G	G	G	G	G	G	G	G	G	G
C447	2.00	7R	7R	10R	10R	45R	65R	70R	75R	GY	20R	20R	20R	GY	GY
C025		Gn	Gn	Gn		Gn	Gn	Gn	Gn	Gn	Gn	Gn	Gn	Gn	Gn
C045		20PHR	25PHR	25PHR		20R	45R	47R	47R	PHR	PHR	PHR	PHR	YSR	YSR

a - These barbed wire numbers do not always correspond to the unfabricated wire numbers as seen opposite:

b - The zinc coating weight reported in this column is the average of all stripping test determinations made on samples of barbed wire of the specific wire lot number.

TABLE II.—*Continued*

		College Station, Texas				Santa Cruz, Calif.				Manhattan, Kan.				Davis, Calif.			
		years exposure				years exposure				years exposure				years exposure			
22.8	23.8	20.9	21.9	22.9	23.9	20.8	21.8	22.9	23.7	20.7	21.8	22.8	23.8	20.8	21.8	22.9	23.8
		100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R
		100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R
		68R	98R	90R	100R	95R	100R	100R	100R	80R	100R	100R	100R	5R	8R	15R	20R
		50R	100R	100R	100R	73R	90R	95R	100R	95R	100R	100R	100R	8R	10R	15R	20R
		10R	15R	70R	70R	20R	20R	28R	25R	5R	30R	60R	60R	Y	Y,SR	Y,SR	Y,SR
		28R	28R	35R	50R	25R	25R	30R	40R	20R	80R	80R	80R	5R	10R	10R	10R
		5R	6R	10R	15R	20R	20R	20R	20R	Y	Y	GY	GY	Y	Y	Y	Y,SR
50R	60R	16R	11R	20R	40R	25R	25R	25R	25R	Y	10R	50R	50R	5R	10R	10R	10R
100R	100R	GY	1R	1R	2R	G	G	G	G	G	G	MG	MG	G	G	G	G
90R	95R	GY	GY	GY	2R	GY	GY	GY	GY	G	G	MG	MG	G	G	G	G
100R	100R	GY	GY	GY	GY	Y	Y	Y	Y	GY	GY	G	G	GY	GY	GY	GY
20R	40R	GY	GY	GY	1R	GY	GY	GY	GY	G	G	MG	MG	GY	GY	GY	GY
Y	Y	G	G	GY	GY	GY	GY	GY	GY	G	G	MG	MG	GY	GY	GY	GY
Y	Y	G	G	G	G	GY	GY	GY	GY	G	G	MG	MG	GY	GY	GY	GY
G	G	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G	G	G
Y	Y	GY	GY	13R	20R	GY	GY	Y	Y	GY	GY	G	G	GY	GY	GY	GY
Y	Y	G	GY	G,RB	G,RB	G	G	GY	GY	G	G	MG	MG	G	G	G	G
G	G	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G	G	G
G	G	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G	G	G
Y	Y	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G	G	G
Gn	Gn	Br	Br	Br	Br	Gn	Gn	Gn	Gn B,100R	Bl	Bl	Bl	BR	BR	BR	BR	Gn
YSR	YSR	5R	8PHR	14PHR	15PHR	33SR	40SR	40SR	40SR	10R	27R	15PHR	15PHR	25SR	25SR	25SR	25SR

Barbed Wire	Unfabricated Wire	Barbed Wire	Unfabricated Wire
C410	410	C441	442
C411	None	C442	441
C412	412	C443	443
C421	None	C445[c]	None
C422	421	C446	None
C420	423	C447	None
C430	431	C450[d]	None
C432	432	C451	451
C431	430	C494	None
		C452	450

c - Aluminum barbs

d - Galvanized after fabrication

TABLE III.—REPORT OF 1957, 1958, 1959 AND 1960 INSPECTIONS OF FARM FIELD FENCE SPECIMENS.

Abbreviations and symbols used to designate appearance or state of corrosion: M = metallic; G = gray; Y = yellowed or rust-stained, but not showing actual rough rust of base metal; R = rough rust of base metal, figures preceding R (as 5, 20, 50, 100) are percentages; PHR, PHY = rusted or yellowed in pinholes; SR, SY = speckled appearance of rust or yellowing; Br, Gn, respectively, signify corroded to a brown or green appearance; MG = intermediate between M and G; GY = predominantly gray but showing indication of Y. All vacant blocks indicate 100R.

Fence No.	Weight of Zinc Coating, oz per sq ft of Surface(a)	Lafayette, Ind.(b)				Ithaca, N. Y.				State College, Pa.				Ames, Iowa	
		21.0 yr	22.0 yr	22.9 yr	24.0 yr	21.0 yr	21.9 yr	22.9 yr	23.9 yr	20.3 yr	21.3 yr	22.3 yr	23.3 yr	20.8 yr	21.8 yr
A215	0.25														
A512	0.26														
A411	0.28														
A315	0.29														
A316	0.29														
Ave	0.27														
A412	0.30														
A510	0.31														
A422	0.31														
A423	0.31														
A216	0.31														
A511	0.32														
A520	0.38														
A317	0.38														
A410	0.39														
Ave	0.33														
A326	0.42														
A327	0.45														
A522	0.47														
A225	0.47														
A521	0.48														
A431	0.48														
A228	0.48														
A421	0.49														
A433	0.49														
A227	0.49														
Ave	0.47														
A532	0.50														
A430	0.52														
A325	0.52														
Ave	0.51														
A531	0.60													100R	100R
A432	0.60													65R	80R
A336	0.60													90R	100R
A238	0.60													70R	100R
A335	0.61													90R	100R
A237	0.61													70R	90R
A235	0.61													70R	90R
A337	0.64													100R	100R
A530	0.65													99R	100R
Ave	0.61														
A338	0.70	95R	100R	100R	100R	100R	100R	100R	100R	95R	98R	95R	100R	25R	40R
A236	0.71	100R	100R	100R	100R	100R	100R	100R	100R	95R	95R	95R	95R	1R	10R
A541	0.75	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	60R	95R
A441	0.76	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	20R	100R
A543	0.77	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	70R	90R
A443	0.78	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	60R	100R
A440	0.79	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	70R	90R
Ave	0.75														
A248	0.80	90R	95R	98R	100R	65R	90R	90R	90R	5R	23R	20R	50R	G	G
A245	0.81	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	10R	40R
A346	0.82	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	SR	SR
A542	0.85	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	70R	90R
A442	0.86	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	G	G
Ave	0.83														
A540	0.93	93R	100R	100R	100R	100R	100R	100R	100R	95R	97R	97R	98R	60R	90R
A347	0.95	100R	100R	100R	100R	88R	90R	90R	95R	95R	95R	90R	95R	Y	Y
A348	0.96	85R	95R	90R	100R	10R	40R	45R	60R	G	5R	6R	20R	G	G
A345	0.96	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	SR	10R
A246	0.96	100R	100R	100R	100R	100R	100R	100R	100R	95R	96R	95R	95R	10R	25R
A247	0.98	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	SR	SR
Ave	0.96														
A252	1.71	G	G	G	G	G	G	G	G	G	G	G	G	G	G
A350	1.76	G	G	G	G	G	G	G	G	G	G	G	G	G	G
A352	1.86	5R	5R	3R	5R	1R	1R	2R	SR	1R	2R	2R	5R	G	G
Ave	1.78														
A397	2.72	G	G	G	G	G	G	G	G	G	G	G	G	G	G
A298	2.73	G	G	G	G	G	G	G	G	G	G	G	G	G	G
Ave	2.73														
A001	12-14% Cr, Cold Drawn	G	G	G	G	D	BP	5SR	10SR	G	G	G	G	GSR	GSR
A002	12-14% Cr, Air Quenched	MG	MG	MG	MG	D	D	MG	MG	G	G	G	G	G	G
A003	18% Cr - 8% Ni	M	M	M	M	M	M	M	M	M	M	MG	MG	M	M
A020	Cu Covered, (7.42 oz per sq ft)	Gn	Gn	Gn	Gn	Gn	Gn	Gn	Gn	Gn	Gn	Gn	Gn	Gn	Gn
A040	Pb Covered, (1.48 oz per sq ft)	30PHR	35PHR	35PHR	35PHR	30R	40R	45R	48R	PHR	PHR	100PHR	100PHR	YSR	YSR

TABLE III.—*Continued*

		College Station, Texas				Santa Cruz, Calif.				Manhattan, Kan.				Davis, Calif.			
22.8 yr	23.8 yr	20.9 yr	21.9 yr	22.9 yr	23.9 yr	20.8 yr	21.8 yr	22.9 yr	23.7 yr	20.7 yr	21.8 yr	22.8 yr	23.8 yr	20.8 yr	21.8 yr	22.9 yr	23.8 yr
						100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R
						98R	98R	98R	100R	100R	100R	100R	100R	100R	100R	100R	100R
						100R	100R	100R	100R	100R	100R	100R	100R	80R	90R	97R	100R
						100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R
						95R	100R	100R	100R	80R	80R	85R	85R	45R	60R	75R	75R
		100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	70R	80R	90R	90R
		100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	68R	80R	95R	100R
		98R	95R	95R	95R	95R	100R	100R	100R	95R	100R	90R	90R	48R	78R	80R	90R
		100R	100R	100R	100R	80R	95R	95R	100R	50R	60R	65R	50R	10R	25R	35R	50R
		100R	100R	100R	100R	95R	100R	100R	100R	90R	100R	100R	100R	25R	50R	55R	60R
		100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R
		100R	100R	100R	100R	75R	90R	95R	100R	50R	60R	100R	90R	40R	60R	70R	70R
		50R	60R	68R	70R	60R	73R	80R	100R	40R	40R	75R	75R	10R	18R	20R	25R
		100R	100R	100R	100R	90R	98R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R
		60R	60R	75R	85R	70R	80R	80R	90R	30R	75R	60R	80R	2R	10R	20R	25R
		6R	10R	15R	25R	18R	25R	40R	40R	Y	Y	10R	G	Y	Y	Y	Y,SR
		53R	55R	75R	75R	40R	50R	65R	90R	Y	Y	30R	20R	8R	20R	30R	30R
		5R	8R	15R	15R	18R	20R	20R	25R	Y	Y	G	G	Y	Y	Y	Y
		68R	90R	95R	100R	73R	81R	90R	90R	50R	50R	65R	75R	45R	55R	55R	55R
		8R	18R	33R	35R	10R	18R	30R	30R	Y	Y	Bl	G	Y	Y	Y	Y,SR
		13R	23R	28R	30R	30R	33R	45R	50R	Y	5R	20R	20R	Y	Y,SR	Y,SR	Y,SR
		45R	50R	55R	70R	45R	60R	80R	80R	25R	25R	Y	25R	23R	25R	25R	25R
		15R	18R	25R	30R	15R	15R	15R	20R	Y	Y	15R	15R	Y	Y	Y	Y
		23R	30R	45R	40R	38R	45R	55R	60R	10R	15R	30R	25R	Y,SR	1R	2R	5R
		6R	13R	38R	45R	20R	20R	30R	30R	Y	Y	Bl,Y	Bl,Y	Y	Y	Y	Y
		8R	10R	18R	20R	15R	15R	20R	20R	Y	Y	10R	GY	Y	Y	Y	Y,SR
		10R	15R	20R	30R	25R	25R	25R	30R	Y	10R	40R	20R	Y	Y	Y	Y
100R	100R	6R	10R	15R	20R	20R	20R	10R	10R	GY	GY	50R	50R	GY	5R	13R	15R
100R	100R	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G	G	G
100R	100R	8R	10R	10R	10R	25R	28R	28R	30R	Y	Y	GY	G	Y	Y	Y	Y,SR
100R	100R	13R	15R	18R	15R	28R	28R	40SR	15R	Y	Y	GY	GY	Y	Y	Y	Y
100R	100R	1R	4R	8R	5R	Y,SR	Y,SR	1R	2R	GY	GY	GY	GY	GY	GY	GY	Y,SR
95R	95R	3R	4R	10R	10R	Y,SR	Y,SR	10SR	10SR	GY	GY	G	G	GY	GY	GY	Y,SR
90R	95R	15R	18R	18R	15R	18R	20R	20R	25R	Y	Y	GY	GY	Y,SR	Y,SR	Y,SR	Y,SR
100R	100R	10R	15R	15R	20R	20R	23R	20R	20R	Y	Y	GY	GY	Y	Y	Y	Y,SR
100R	100R	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G	G	5R
50R	60R	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G	G	G
10R	20R	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G	G	G
95R	100R	1R	5R	13R	10R	G	GY	GY	GY	GY	GY	GY	GY	GY	GY	GY	G,SR
100R	100R	GY	GY	3R	3R	GY	GY	GY	GY	GY	G	MG	MG	G	G	G	G,SR
100R	100R	1R	1R	4R	2R	GY	GY	GY	GY	GY	GY	MG	MG	GY	GY	GY	G,SR
100R	100R	GY	GY	GY	GY	G	GY	GY	SR	GY	GY	GY	G	GY	GY	GY	Y,SR
90R	95R	GY	GY	3R	3R	G	GY	GY	GY	GY	GY	MG	MG	GY	G	G	G,SR
G	G	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G	G	G
50R	75R	GY	2R	4R	5R	Y,SR	Y,SR	Y,SR	Y,SR	GY	GY	G	G	GY	GY	GY	GY
10R	10R	GY	GY	3R	3R	Y	8R	Y,SR	Y,SR	GY	GY	MG	G	GY	GY	GY	GY
95R	95R	1R	3R	5R	5R	GY	GY	1R	1R	GY	GY	Y	Y	GY	GY	GY	GY
G	G	G	GY	GY	GY	G	G	G	G	G	G	MG	MG	G	GY	G	G
90R	95R	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G	G	G
10R	15R	GY	1R	1R	1R	16R	16R	15R	15R	GY	G	MG	MG	G	G	G	G
G	G	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G	G	G
15R	20R	3R	8R	8R	5R	13R	13R	10R	10R	Y	Y	GY	G	Y	Y	Y	Y,SR
30R	40R	GY	1R	4R	3R	GY	GY	GY	GY	GY	GY	MG	G	G	G	GY	GY
SR	SR	13R	13R	13R	15R	8R	10R	10R	15R	Y	Y	Y	Y	Y	Y	Y	Y,SR
G	G	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G	G	G
G	G	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G	G	G
G	G	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G	G	GY
G	G	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G	G	G
G	G	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G	G	G
GSR	G,SR	13R	18R	23R	20R	10R	13R	8R	15R	G,SR	G,SR	18R	10R	25R	25R	25R	30R
G	G	8R	13R	13R	10R	5R	5R	5R	5R	G,SR	G,SR	M,Bl	M	5R	5R	5R	5R
M	M	M,SY	M,SY	M,SY	M,SY	MG	MG	MG	MG	M	M	M	M	MG	MG	MG	MG
Gn	Gn	Br	Br	Br	Br	Gn	Gn	Gn	Gn	Bl	Bl	Bl	BR	BR	BR	BR	BR
YSR	YSR	5R	15PHR	18PHR	15PHR	10R	8R	8R	10R	40R	40R	20PHR	20PHR	1R	2R	2R	2R

TABLE IV.—REPORT OF 1957, 1958, 1959 AND 1960 INSPECTIONS OF CHAIN LINK FENCE SPECIMENS.

Abbreviations and symbols used to designate appearance or state of corrosion: M = metallic; G = gray; Y = yellowed or rust-stained, but not showing actual rough rust of base metal; R = rough rust of base metal, figures preceding R (as 5, 20, 50, 100) are percentages; PHR, PHY = rusted or yellowed in pinholes; Br, Gn, respectively, signify corroded to a brown or green appearance; SR, SY = speckled appearance of rust or yellow; MG = intermediate between M and G; GY = predominantly gray but showing indication of Y. All vacant blocks indicate 100R.

All 200 series wires are 9 gage. Lead-coated wire, 9 gage. Corrosion-resistant steel wire, 14 gage.

All 100 series wires are 6 gage. Copper-covered wire, 0.128 in. diameter.

Average Weight of Zinc Coating, oz per sq ft of surface	Fence No.	Bridgeport, Conn.				Lafayette, Ind.				Ithaca, N. Y.				State College, Pa.				Santa Cruz, Calif				Davis, Calif.			
		Years Exposure				Years Exposure				Years Exposure				Years Exposure				Years Exposure				Years Exposure			
		20.4	21.4	22.4	23.4	21.0	22.0	22.9	24.0	21.0	21.9	22.9	23.9	20.3	21.3	22.3	23.3	20.8	21.8	22.9	23.7	20.8	21.8	22.9	23.8
0.29	E280^{a}	*																100R	100R	100R	100R	85R	90R	95R	100R
0.52	E281	*																10R	15R	20R	20R	Y	Y	Y	Y, SR
0.37	E282	*																30R	40R	65R	75R	20R	23R	25R	25R
1.22	E185	*				95R	98R	99R	99R	100R	100R	100R	100R	75R	99R	100R	100R	G	G	G	GG	GY	GY	GY	GY
1.13	E285	*				100R	100R	100R	100R	96R	96R	96R	97R	98R	99R	100R	100R	G	G	G	G	GY	GY	GY	GY
2.92	E290	Y	60R	-	90R	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G
2.09	E291	10R	60R	-	90R	G	G	G	G	1R	1R	1R	1R	G	G	G	5R	G	G	G	G	G	G	G	G
1.60	H280^{a}	*				85R	90R	95R	95R	80R	85R	90R	90R	60R	67R	50R	80R	G	G	G	G	G	G	G	G
1.83	H185	*				95R	98R	98R	99R	100R	93R	95R	95R	40R	55R	75R	85R	G	G	G	G	GY	GY	GY	GY
1.60	H186	*				85R	90R	90R	95R	85R	85R	85R	85R	25R	55R	50R	60R	G	G	G	G	G	G	G	G
1.61	H187	*				45R	50R	60R	70R	10R	23R	30R	33R	GY	GY	GY	5R	G	G	G	G	G	G	G	G
1.55	H188	*				95R	95R	95R	96R	90R	100R	100R	100R	40R	45R	60R	75R	G	G	G	G	GY	GY	GY	GY
1.36	H285	*				85R	90R	90R	95R	97R	97R	98R	98R	98R	65R	80R	90R	G	G	G	G	G	G	G	G
1.97	H286	*				65R	75R	80R	80R	100R	100R	100R	100R	20R	50R	40R	60R	GY	GY	GY	GY	GY	GY	GY	GY
1.46	H287	*				80R	80R	80R	80R	90R	90R	95R	95R	80R	85R	80R	90R	G	G	G	G	G	G	G	G
1.74	H288	*				60R	75R	80R	80R	23R	23R	35R	48R	Y	1R	1R	10R	G	G	G	G	GY	GY	GY	GY
1.40	H289^{b}	*				G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G
4.21	H290	Y	100R	-	100R	1R	1R	GY	GY	25R	85R	88R	88R	GY	GY	Y	Y	MG	MG	MG	G	G	G	G	G
Copper Covered (8.52 oz per sq ft)	K030	GN	C	-	-	GN	GN	GN	GN	GN	GN	GN	GN	GN	GN	GN	GN	BR	BR	BR	BR	BR	BR	BR	BR
Lead Coated (1.75 oz per sq ft)	L050	B	5R	-	5R	55PHR	55PHR	40PHR	40PHR	18R	18R	25R	28R	PHR	PHR	PHR	100R&Y	35SR	50SR	65SR	70SR	23SR	23SR	23SR	23SR
Corrosion Resistant Steel (18% Cr, 8% Ni)	J011	MG	MG	-	MG	MG	MG	M	M	M	M	M	M	M	M	MG	MG	MG	MG	MG	MG	MG	MG	MG	MG

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[a] E numbers, coating applied before weaving; H numbers, coating applied after weaving.

[b] Although this sample has an H number, its appearance indicates that it was galvanized before weaving.

[c] Stolen between inspection on 4/23/57 and 4/16/58.

* Specimens destroyed shortly after the 16.5 yr inspection, all 100R.

TABLE V.—REPORT OF 1957, 1958, 1959 AND 1960 INSPECTIONS OF WIRE STRAND SPECIMENS.

Abbreviations and symbols used to designate appearance or state of corrosion: M = metallic; G = gray; Y = yellowed or rust-stained, but not showing actual rough rust of base metal; R = rough rust of base metal, figures preceding R (as 5, 20, 50, 100) are percentages; PHR, PHY = rusted or yellowed in pinholes; Br, Gn, respectively, signify corroded to a brown or green appearance; SR, SY = speckled appearance of R or Y; MG = intermediate between M and G; GY = predominantly gray but showing indication of Y.

Average Weight of Zinc Coating, oz per sq ft of Surface	Strand No.	Bridgeport, Conn.				Lafayette, Inc.				Ithaca, N. Y.				State College, Pa.			
		20.4 yr	21.4 yr	22.4 yr	23.4 yr	21.0 yr	22.0 yr	22.9 yr	24.0 yr	21.0 yr	21.9 yr	22.9 yr	23.9 yr	20.3 yr	21.3 yr	22.3 yr	23.3 yr
0.69	D345	100R	100R	-	100R		43R	35R	43R	60R	50R	65R	78R	2R	5R	10R	50R
0.87	D347	100R	100R	-	100R		78R	78R	83R	40R	20R	28R	40R	18R	35R	13R	75R
0.88	D346	100R	100R	-	100R		48R	40R	45R	3R	6R	9R	15R	3R	6R	8R	30R
0.93	D348	100R	100R	-	100R		50R	53R	73R	17R	28R	35R	53R	1R	1R	1R	4R
1.03	D362	100R	100R	-	100R		68R	68R	73R	15R	30R	35R	40R	4R	6R	8R	18R
1.10	D360	100R	100R	-	100R		25R	23R	25R	G	4R	5R	8R	G	GY	GY	3R
1.17	D361	100R	100R	-	100R		35R	23R	28R	15R	10R	18R	25R	GY	GY	GY	4R
1.32	D367	100R	100R	-	100R		43R	23R	30R	25R	23R	25R	33R	Y	Y	Y	Y
1.39	D366	50R	90R	-	100R		G	G	G	G	G	1R	1R	G	G	G	G
1.51	D365	100R	100R	-	100R		68R	GY	GY	8R	10R	15R	20R	GY	GY	GY	GY
1.71	D371	GY	20R	-	90R		G	G	G	G	G	G	G	G	G	G	G
2.47	D375	GY	GY	-	20R		G	G	G	G	G	G	G	G	G	G	G
Lead Coated (1.96 oz per sq ft)	D045	D	5R	-	5R		50PHR	50PHR	55PHR	28R	55R	70R	78R	PHR	PHR	PHR	100R&Y

Average Weight of Zinc Coating, oz per sq ft of Surface	Strand No.	Ames, Iowa				College Station, Texas				Santa Cruz, Calif.				Manhattan, Kansas				Davis, Calif.			
		20.8 yr	21.8 yr	22.8 yr	23.8 yr	20.9 yr	21.9 yr	22.9 yr	23.9 yr	20.8 yr	21.8 yr	22.9 yr	23.7 yr	20.7 yr	21.8 yr	22.8 yr	23.8 yr	20.8 yr	21.8 yr	22.9 yr	23.8 yr
0.69	D345	GY	G	G	G	G	G	G	G	GY	GY	GY	GY	G	G	G	G	GY	GY	GY	GY
0.87	D347	GY	GY	GY	GY	G	G	GY	GY	GY	GY	GY	GY	GY	GY	G	G	GY	GY	GY	GY
0.88	D346	GY	GY	GY	GY	G	G	G	G	MG	MG	MG	G	G	G	G	G	G	G	G	G
0.93	D348	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G
1.03	D362	Y	Y	Y	Y	G	G	GY	GY	G	G	G	G	GY	GY	G	G	G	GY	GY	GY
1.10	D360	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G
1.17	D361	GY	GY	G	GY	G	G	GY	GY	GY	GY	GY	GY	GY	GY	G	G	GY	GY	GY	GY
1.32	D367	Y	Y	Y	Y	G	G	GY	GY	G	GY	GY	GY	GY	GY	G	G	G	GY	GY	GY
1.39	D366	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G
1.51	D365	GY	GY	GY	GY	G	G	G	G	GY	GY	GY	GY	GY	G	G	G	GY	GY	GY	GY
1.71	D371	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G
2.47	D375	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G
Lead Coated (1.96 oz per sq ft)	D045	YSR	YSR	YSR	YSR	25PHR	25PHR	25PHR	30PHR	68SR	10R	10R	13R	40R	15R	15PHR	18PHR	10R	10R	10R	10R

TABLE VI.—BREAKING LOADS OF WIRES REMOVED AFTER APPROXIMATELY 21 YEARS OF EXPOSURE.

Wire Lot No.	Original Breaking Load, lb	Breaking Load, lb	Loss, Percent	Wire Lot No.	Original Breaking Load, lb	Breaking Load, lb	Loss, Percent
Ithaca, N. Y.							
501	500	269	46.2	423	400	319	20.3
506	520	271	49.8	430	780	645	17.3
507	590	301	49.0	431	880	699	20.6
510	500	310	38.0	432	510	424	16.9
511	280	171	38.9	440	590	514	12.9
512	320	197	38.4	441	520	447	14.0
520	280	209	25.4	442	470	379	19.8
521	270	199	26.3	443	750	633	15.6
522	500	346	30.8	444	570	492	13.7
530	310	209	32.6	495	750	652	13.1
531	500	354	29.2	315	890	759	14.7
540	300	224	25.3	316	1110	938	15.5
541	350	282	19.4	326	700	603	13.9
542	510	361	29.2	327	1160	973	16.1
543	300	247	17.7	335	1040	913	12.2
410	570	448	21.4	336	760	651	14.3
411	630	520	17.5	337	860	780	9.3
412	840	636	24.3	235	1680	1538	8.5
421	470	374	20.4	247	1790	1582	11.6
422	500	416	16.8	115	2690	2470	8.2
003	1280	1280	0.0	015	1250	1240	0.01
008	2510	2565	-0.02	021	1640	1630	0.01
009	290	265	0.09	022	2320	2290	0.01
Lafayette, Ind.							
510	500	264	47.2	431	880	627	28.8
511	280	127	54.6	432	510	379	25.7
512	320	145	54.7	440	590	433	26.6
520	280	170	39.3	442	470	347	26.2
521	270	148	45.2	444	570	435	23.7
522	500	257	48.6	495	750	585	22.0
530	310	169	48.1	315	890	688	22.7
531	500	290	42.0	316	1110	852	23.2
540	300	191	36.3	317	780	635	18.6
541	350	212	39.4	326	700	532	24.0
542	510	292	42.7	327	1160	903	22.2
543	300	199	33.7	329	720	564	21.7
411	630	401	36.3	335	1040	826	20.6
412	840	560	33.3	336	760	608	20.0
421	470	307	34.7	337	860	673	21.7
422	500	363	27.4	345	890	842	5.4
423	400	261	34.8	235	1680	1162	30.8
430	780	555	28.8	021	1640	1630	0.01
				022	2320	2290	0.01
Ames, Iowa							
510	500	397	20.6	022	2320	2310	0.0
511	280	215	23.2	001	1230	1265	-0.03
512	320	243	24.1	004	2420	2330	0.04
522	500	431	13.8	005	320	325	-0.02
530	310	272	12.3	002	1310	1320	-0.01
531	500	430	14.0	006	2480	2530	-0.02
412	840	703	16.3	007	280	295	-0.05
431	880	780	11.4	003	1280	1280	0.0
117	2230	2102	10.2	008	2510	2615	-0.04
021	1640	1635	0.0	009	290	290	0.0
				015	1250	1225	0.02
Manhattan, Kansas							
510	500	518	-3.6	006	2480	2485	0.0
410	570	572	0.0	007	280	290	-0.04
217	1080	1087	0.0	003	1280	1295	-0.01
001	1230	1270	-0.03	008	2510	2540	-0.01
004	2420	2320	0.04	009	290	280	0.03
005	320	325	-0.02	015	1250	1270	-0.02
002	1310	1355	-0.03	021	1640	1645	0.0
				022	2320	2320	0.0

TABLE VI.—*Continued*

Wire Lot No.	Original Breaking Load, lb	Breaking Load, lb	Loss, Percent	Wire Lot No.	Original Breaking Load, lb	Breaking Load, lb	Loss, Percent
Santa Cruz, California							
510	500	454	9.2	008	2510	2545	-0.01
511	280	242	13.6	009	290	280	0.03
512	320	266	16.9	021	1640	1650	-0.01
003	1280	1275	0.0	022	2320	2325	0.0
College Station, Texas							
512	320	273	14.7	009	290	280	0.03
410	570	526	7.8	015	1250	1270	-0.02
003	1280	1275	0.0	021	1640	1640	0.0
008	2510	2540	-0.01	022	2320	2310	0.0
Davis, California							
003	1280	1290	-0.01	021	1640	1635	0.0
008	2510	2450	0.02	022	2320	2315	0.0
009	290	270	0.07				
State College, Pa.							
530	310	170	45.2	495	750	630	16.0
531	500	250	50.0	326	700	530	24.3
540	300	195	35.0	327	1160	900	22.4
541	350	230	34.3	329	720	590	18.0
542	510	315	38.2	335	1040	830	20.2
543	300	201	33.0	336	760	610	19.7
430	780	560	28.2	337	860	740	14.0
431	880	610	30.7	338	1020	860	15.7
432	510	392	23.1	345	890	710	20.2
440	590	437	25.9	346	1070	870	28.0
441	520	402	22.7	347	860	725	15.7
442	470	345	26.6	245	1570	1355	13.7
443	750	575	23.3	247	1790	1610	10.0
444	570	445	21.9	248	1450	1280	11.7

Findings from Tension Tests:

Tension tests are made only on the unfabricated wire specimens. These specimens are exposed in groups of seven taken from the same lot of wire and supposedly alike. An attempt is made to remove and test the first specimen of each group when it has been estimated to have lost 5 to 10 per cent of its strength, and the seventh specimen when it is estimated to have lost some 75 per cent of its strength. There were originally about 840 tensile strength specimens exposed at each site. The number of specimens removed to date is shown in Table B.

The results of the tensile strength tests of the specimens removed from 1957 to 1960 are included in Table VI.

Respectfully submitted on behalf of the Wire Test Inspection Committee,

FRED M. REINHART,
Chairman

APPENDIX III

REPORT OF SUBCOMMITTEE XVI ON FIELD TESTS OF ATMOSPHERIC CORROSION OF HARDWARE

1958 Program

This report covers the 1960 inspection of coated hardware specimens exposed in 1958 at Newark, N. J. and at the 80- and 800-ft sites at Kure Beach, N. C.

The hardware specimens remaining from the 1929 program were inspected at State College, Pa., on April 19, 1960. The results showed little change since the 1959 inspection reported in the 1960 ASTM *Proceedings*, p. 116, and are therefore not included in this report.

Background and details of the 1958 Hardware Program may be found in the 1959 ASTM *Proceedings*, p. 133. These include basis metal compositions, coating procedures, individual coating thicknesses, and microscopic examinations. In the 1960 ASTM *Proceedings* are condensed tabulations of the ranges and averages of thickness for all sets of specimens together with the one year inspection report.

In Tables I, II, and III, attached, are the 2-yr inspection data for the Newark and Kure Beach (80- and 800-ft site) specimens. Only the specimens showing rust are itemized in the tables; all others show none. The numbering system for specimen identification is shown in Fig. 2 of the report in the 1959 ASTM *Proceedings*.

None of the rusting observed to date is advanced, and suggested patterns must be regarded as tentative. Sprayed aluminum (coating No. 10) and sprayed zinc (coating No. 9) show no rust at any location other than the slight rust staining around identification holes of coating No. 10 reported earlier. Of the 31 specimens showing 5 per cent or more rust, 23 are coated with hot-dipped aluminum (coating No. 7), 7 are coated with hot-dipped zinc (coating No. 5) and 1 with electroplated zinc (coating No. 8). Of these 31 specimens showing 5 per cent or more rust, 18 are bent, 12 are flat, and 1 is round.

Table IV presents weight loss data for uncoated panels of six of the basis steels removed from the three sites after approximately two years exposure. These weight losses of the basis steels reflect an initially high corrosion rate which, particularly in the case of the low-alloy steels, becomes much reduced with increased exposure. Corrosion rates have not been calculated from these data since they would be misleading if extrapolated to periods greater than two years.

Respectfully submitted on behalf of the subcommittee,

W. W. Bradley,
Chairman.

TABLE I.—INSPECTION OF COATED STEEL HARDWARE SPECIMENS AT NEWARK, N. J.

Inspected 5–13–60; exposure period 1.92 years.

Abbreviations and symbols used to designate appearance or state of corrosion: 0 = no rust; 5, 10, 25, 100 = approximate percentages of rust; R = rusting of base metal; Y = yellow or orange rust; R* = rust spots widely distributed; Tr = trace.

Steel Coating Location Replicate	Flat Panel			Bent Panel			Rounds	
	Coating Thickness, mils	Topside	Under Side	Coating Thickness, mils	Topside	Under side	Coating Thickness, mils	Sides
Hot-dip zinc (Code 5)								
0–5–11–15	[a]							
0–5–11–16	...	...	...	2.18	...	1 spot		
1–5–11–15	2.24	1 rust spot	...	...	...	...		
2–5–11–14	3.66	20Y	...	...	...	...		
2–5–11–15	2.81	15Y	...	...	...	...		
2–5–11–16	3.14	45Y	...	2.29	40Y	...		
2–5–11–17	3.08	5Y	...	...	...	...		
2–5–11–18	3.74	15Y	...	2.28	30Y	...		
3–5–11–16	2.86	...	1 rust spot	...	...	...		
Hot-dip aluminum (Code 6)								
0–6–11–18	2.51	TrY	...	...	...	...		
2–6–11–17	2.06	TrY	...	...	...	...		
3–6–11–18	2.57	TrY	...	1.59	...	TrR		
4–6–11–14	2.66	TrR, TrY	...	...	...	...		
Hot-dip aluminum (Code 7)								
0–7–11–16	...	...	...	...	...	...	2.15	TrY
0–7–11–17	...	...	...	...	...	...	2.87	TrY
1–7–11–15	...	...	...	...	...	...	3.11	TrY
1–7–11–16	...	...	...	...	...	...	3.26	TrY
1–7–11–17	...	...	...	...	...	...	2.98	TrY
3–7–11–15	...	...	...	2.45	TrY	...		
4–7–11–14	...	...	...	2.40	TrY	TrY		
4–7–11–18	...	...	...	2.32	...	TrR, TrY		
Electroplated zinc (Code 8)								
1–8–11–16	...	...	...	...	...	...	1.64	TrR
1–8–11–18	2.0	...	TrR	...	...	...		
Sprayed aluminum (Code 10)	[b]							

[a] Removed because of damage.

[b] Light rust staining around identification holes.

TABLE II.—INSPECTION OF COATED STEEL HARDWARE SPECIMENS AT KURE BEACH (800-FT SITE), N. C.

Inspected 5–31–60; exposure period 1.94 years.

Abbreviations and symbols used to designate appearance or state of corrosion: 0 = no rust; 5, 10, 25, 100 = approximate percentages of rust; R = rusting of base metal; Y = yellow or orange rust; R* = rust spots widely distributed; Tr = trace.

Steel Coating Location Replicate	Flat Panel			Bent Panel			Rounds	
	Coating Thickness, mils	Topside	Under Side	Coating Thickness, mils	Topside	Underside	Coating Thickness, mils	Sides
Hot-dipped aluminum (Code 6)								
0–6–12–14	...	...	...	1.98	...	TrY		
2–6–12–15	...	...	...	1.86	...	TrY		
3–6–12–14	2.46	TrY	...	...	...	...		
3–6–12–17	2.43	TrY	TrY	...	...	...		
4–6–12–17	...	...	...	1.89	TrY	TrY		
4–6–12–18	...	...	...	2.21	...	TrY		
Hot-dipped aluminum (Code 7)								
0–7–12–14	2.40	TrR	5R	2.10	TrY	...	1.06	20R
0–7–12–16	...	...	...	2.02	TrY	TrY		
0–7–12–18	...	...	...	2.32	...	10*		
1–7–12–15	3.79	TrR	...	...	...	...		
2–7–12–14	3.33	25R*	30R*	...	...	...		
2–7–12–15	...	...	...	2.28	...	TrY		
2–7–12–16	3.79	30R	20R, 25R*	2.96	TrY	TrY		
2–7–12–17	3.68	TrR	...	2.52	10Y	30Y		
2–7–12–18	...	...	...	2.60	TrY	TrY		
3–7–12–14	...	...	...	2.49	5Y	5Y		
3–7–12–15	...	...	...	2.45	5Y	5Y		
3–7–12–16	2.87	TrR	5*	2.06	5Y	5Y		
3–7–12–17	...	...	...	1.90	TrY	5Y		
3–7–12–18	...	...	...	3.41	...	TrY		
4–7–12–14	...	...	...	2.59	TrY	...		
4–7–12–15	...	...	...	2.80	TrY	...		
4–7–12–16	...	...	...	2.39	TrY	TrY		
4–7–12–17	...	...	...	2.46	TrY	...		
4–7–12–18	...	...	...	2.76	TrY	...		
Sprayed aluminum (Code 10)	[a]							

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[a] Light rust staining around identification holes.

TABLE III.—INSPECTION OF COATED HARDWARE SPECIMENS AT KURE BEACH (80-FT SITE), N. C.

Inspected 5–31–60; exposure period 1.94 years.

Abbreviations and symbols used to designate appearance or state of corrosion: 0 = no rust; 5, 10, 25, 100 = approximate percentages of rust; R = rusting of base metal; Y = yellow or orange rust; R* = rust spots widely distributed; Tr = trace.

Steel Coating Location Replicate	Flat Panel			Bent Panel			Rounds	
	Coating Thickness, mils	Topside	Underside	Coating Thickness, mils	Topside	Underside	Coating Thickness, mils	Sides
Hot-dip aluminum (Code 6)								
0–6–13–15	2.77	TrY	. . .					
0–6–13–17	2.08	TrY	. . .					
0–6–13–18	2.58	TrY	. . .					
2–6–13–14	. . .	. . .	. . .	1.74	TrY	. . .		
3–6–13–16	2.33	TrY	. . .					
4–6–13–16	. . .	. . .	. . .	2.29	TrY	. . .		
4–6–13–18	2.43	TrY	. . .					
Hot-dip aluminum (Code 7)								
0–7–13–14	. . .	. . .	. . .	1.69	10R, 10Y	20Y		
0–7–13–15	3.33	TrY		1.84	10R, TrY	TrY		
0–7–13–16	. . .			1.91	TrY	. . .		
0–7–13–17	. . .			2.04	20R, 5Y	10Y		
0–7–13–18	3.12	TrY	TrY	1.86	10Y	TrY		
1–7–13–14	2.28	5Y	TrY	2.29	. . .	TrY		
2–7–13–14	3.01	20R, 10Y	55R	2.21	25R, 5Y	30Y		
2–7–13–15	. . .	. . .	. . .	3.30	TrY	TrY		
2–7–13–16	. . .	. . .	. . .	2.30	5R, TrY	20Y		
2–7–13–17	. . .	. . .	. . .	3.31	TrY	TrY		
2–7–13–18	. . .	. . .	. . .	2.25	10R, 20Y	5Y		
3–7–13–14	. . .	. . .	. . .	2.26	10R	5Y		
3–7–13–15	. . .	. . .	. . .	2.35	5Y	TrY		
3–7–13–16	. . .	. . .	. . .	2.36	TrY	TrY		
3–7–13–17	. . .	. . .	. . .	2.40	TrY	TrR, 5Y		
3–7–13–18	2.84	TrY	. . .	2.16	TrR	TrY		
Electroplated zinc (Code 8)								
1–8–13–18	2.53	. . .	15R					

TABLE IV.—WEIGHT LOSS OF UNCOATED BASIS METALS.

Alloy	Newark, N. J. 1.92 yr Weight, g			Kure Beach (80-ft site) N.C. 1.70 yr Weight, g			Kure Beach (800-ft site), N.C. 1.70 yr Weight, g		
	Original	Final[a]	Loss	Original	Final[a]	Loss	Original	Final[a]	Loss
Code 1—Carbon steel	805.4	792.5	12.9	824.3	802.2	22.1	808.6	794.6	14.0
Code 21—Malleable iron	717.3	703.8	13.5	703.2	682.1	21.1	736.0	723.4	12.6
Code 2—Low alloy steel I	750.0	742.8	7.2	769.9	757.7	12.2	751.0	742.2	8.8
Code 3—Low alloy steel II	741.4	735.4	6.0	738.3	724.9	13.4	736.2	727.0	9.2
Code 4—Low alloy steel III	765.8	756.2	9.6	742.4	729.1	13.3	756.2	745.9	10.3
Code 0—Low alloy steel IV	776.1	771.5	4.6	775.0	763.1	11.9	768.2	759.5	8.7

[a] After stripping of corrosion products.

REPORT OF COMMITTEE A-6
ON
MAGNETIC PROPERTIES*

Committee A-6 on Magnetic Properties held two general meetings during the year: on June 28, 1960, during the Annual Meeting of the Society in Atlantic City, N. J., and on Jan. 17, 1961, in Pittsburgh, Pa. Meetings of several subcommittees and task groups were held concurrently with the general meetings.

The present membership of the committee is 36, of whom 16 are classified as producers, 16 as consumers, and 4 as general interest members. The committee membership increased by 1 during the year, and there were 3 replacements. There have been no changes in the basic organization of the committee.

RECOMMENDATION ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1960 Annual Meeting, Committee A-6 presented to the Society through the Administrative Committee on Standards the recommendation for the revision of the Standard Definitions of Terms, with Symbols, Relating to Magnetic Testing (A 340 – 49), and reversion of the definitions to tentative. This recommendation was accepted by the Standards Committee on May 10, 1961, and the definitions appear in the 1961 Book of ASTM Standards, Part 3.

STANDARDS CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following standards:

Standard Methods of:

Testing Magnetic Materials (**A 34 – 55**), and
Test for Normal Induction and Hysteresis of Magnetic Materials (**A 341 – 55**).

Standard Specifications for:

Flat-Rolled Electrical Steel (**A 345 – 55**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[1]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Definitions (I. L. Cooter, chairman) has revised definitions of terms and symbols maintaining uniformity between ASTM definitions and the definitions of the American Standards Association Sectional Committee C42.

Subcommittee II on Methods of Sampling and Test (J. W. Hale, chairman) has been continuing work on Specification A 343 with particular emphasis on matters pertaining to precision of test and accuracy of test methods.

The Task Group on the Effect of Aluminum Additions to Silicon Steel on Silicon-Steel Density is still collecting information on material with and without oxide coatings for revision of portions of Method A 34.

The Task Group on D-C Testing of High Coercive Magnets is currently circulating a series of magnets for interlaboratory tests. Considerable progress has been made in the preparation of the Manual on Non-Standard Methods of Magnetic Testing which will also include some basic information on the underlying facts of magnetic testing, and the task group has expressed the hope that it will soon be ready for publication.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Although much of the work of the committee in the past has dealt with silicon steel and problems relating to it, there has been a considerable increase in the activity on the special alloys of the nickel-iron variety which are becoming of increasing commercial importance.

This report has been submitted to letter ballot of the committee, which consists of 36 voting members; 30 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

A. C. BEILER,
Chairman.

W. S. EBERLY,
Secretary.

REPORT OF COMMITTEE A-7
ON
MALLEABLE IRON CASTINGS*

Committee A-7 on Malleable Iron Castings held two meetings during the year: on June 28, 1960, at Atlantic City, N. J., and on Jan. 31, 1961, at Cincinnati, Ohio. A number of subcommittees met on the same occasions.

The committee consists of 43 members, of whom 20 are classified as producers, 10 as consumers, and 13 as general interest members.

A Report on Corrosion Test Results on 15 Ferrous Metals After One Year Atmospheric Exposure, prepared by Gordon B. Mannweiler, Chairman of Subcommittee VII on Corrosion, is appended hereto.[1]

REVISION OF TENTATIVE

The committee recommends revisions as follows of the Tentative Specifications for Pearlitic Malleable Iron Castings (**A 220 - 55 T**):[2]

Section 1.—Add to the end of the scope "at both normal and elevated temperatures" and a new note to read as follows:

> NOTE.—For continuous service of some pearlitic malleable irons at elevated temperatures, limiting stress levels are suggested in the Appendix to this specification.

Section 3.—Change the title from "Physical Properties" to read "Tensile Properties." In Paragraph (c), change "L48005" to read "L53004."

Section 4(a).—Transpose the two sentences.

Table I.—In both places change the hardness value of "228" to read "229."

Appendix.—Add a new Appendix I on Elevated Temperature Characteristics, as appended hereto.[3]

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

Standard Specifications for Malleable Iron Castings (A 47 - 52):[2]

Section 1.—Add to the end of the scope "at both normal and elevated temperatures" and a new note to read as follows:

> NOTE.—For continuous service of some malleable irons at elevated temperatures, limiting stress levels are suggested in the Appendix to this specification.

Appendix.—Add a new Appendix I on Elevated Temperature Characteristics, as appended hereto.[3]

Standard Specifications for Malleable Iron Flanges, Pipe Fittings, and Valve Parts for Railroad, Marine, and Other Heavy Duty Service (A 338 - 54):[2]

Title.—Add to the end of the title "at temperatures up to 650 F."

Section 1.—Add to the end of the

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] See p. 164.

[2] 1958 Book of ASTM Standards, Part 1.

[3] The new Appendix appears in the revised specifications; see 1961 Book of ASTM Standards, Part 1.

scope, "Service shall include up to 650 F.[3]"

REAPPROVAL OF STANDARD

The committee recommends the reapproval of the Standard Specification for Cupola Malleable Iron (**A 197 – 47**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

This report has been submitted to letter ballot of the committee, which consists of 43 members; 27 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

CARL F. JOSEPH,
Chairman.

JAMES H. LANSING,
Secretary.

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[3] Based on ASTM Technical Paper, "Stress Rupture Properties of Malleable Iron at Elevated Temperatures," by Lauriston C. Marshall and George F. Sommer, *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 733 (1958).

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

APPENDIX

CORROSION TEST RESULTS ON 15 FERROUS METALS AFTER 1-YR ATMOSPHERIC EXPOSURE

REPORT OF SUBCOMMITTEE VII ON CORROSION

SYNOPSIS

Corrosion test data are presented for 15 different ferrous materials after a 1-yr exposure to the atmosphere at five different locations. These comprise the first series of four removals over a 12-yr period. Weight loss comparisons between uncoated cast surfaces and uncoated machined surfaces are given for a dozen cast metals. Malleable irons, in general, were found to resist atmospheric attack decidedly better than did unalloyed rolled steel. Lower corrosion rates were achieved by alloying with small amounts of copper (or other alloys). Rural sites seemed to be the least severe on the unprotected metals. The cast (graphitic) materials showed greater attack at industrial sites, whereas marine locations proved to be harder on the wrought steels.

In the fall of 1955, through the persevering efforts of the late William A. Kennedy, then chairman of ASTM Committee A-7 on Malleable Iron Castings, and James H. Lansing, Secretary of Committee A-7, plans were initiated to expose several grades of standard (ferritic) malleable and pearlitic malleable irons to prevailing atmospheres at several different sites. The full cooperation of the ASTM Advisory Committee on Corrosion made possible the exposure of the test materials at established ASTM exposure sites throughout the country. Some $2\frac{1}{2}$ yr were consumed in planning the program and in obtaining, identifying, machining, cleaning, and weighing the 1620 specimens. In the early summer of 1958 all (uncoated) test plates were set out at five different sites. Removal of a representative one fourth of all plates and subsequent evaluation of the extent of corrosion were scheduled after completion of 1, 3, 7, and 12 yr exposure. This report covers the subcommittee's findings after the first year's exposure.

TEST MATERIALS

In order to be certain that the tests would be representative of the product of the malleable industry as a whole, 10 different malleable irons were selected. These were produced by leading commercial foundries in accordance with ASTM Specifications A 47 – 52[1] and A 220 – 55 T.[2] For the sake of comparison, in addition to the malleable irons, a series of each of the two grades of nodular iron (complying with ASTM Specification A 339 – 55[3]) and three rolled steels were included among the materials for testing. Cast steels were not chosen for this investigation because under other auspices a number of cast steels were already under test at the Kure Beach and East Chicago sites, starting in October 1953 for 1-, 3-, and 7-yr periods. The inclusion of gray iron specimens was considered, but advice from Committee A-3 on Cast Iron indicated that there was a lack of interest in the atmospheric corrosion-resistance of gray irons. A listing of the 15 metals exposed under this program is given in Table I.

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[1] Standard Specification for Malleable Iron Castings (A 47 – 52), 1958 Book of ASTM Standards, Part 1, p. 1475.

[2] Tentative Specifications for Pearlitic Malleable Iron Castings (A 220 – 55 T), 1958 Book of ASTM Standards, Part 1, p. 1480.

TABLE I.—BASIS METALS FOR TEST.

Material	Manufacturing Process	Code	Specimen Identification	
			Letter, First Hole	Number of Notches
Standard malleable iron, ASTM A 47 – 52, grade 35018	cold-melt, air-furnace	35018 M.I.—(L)	A	0
		35018 M.I.—(E)	B	1
Alloyed malleable iron, containing 0.5 to 0.75 per cent copper	cold-melt, air-furnace (same heat as one above)	35018 M.I. + Cu	C	2
Standard malleable iron, ASTM A 47 – 52, grade 32510	duplex, cupola-air furnace	32510 M.I.—(N)	D	3
		32510 M.I.—(A) (Hi Si)	E	4
Pearlitic malleable iron, ASTM A 220 – 55 T, grade 45010	cold - melt, air - furnace; reheat, quench and temper	45010 PMI—(L)	F	5
	cold - melt, air - furnace; Mn-alloyed, air-quench and temper	45010 PMI—Mn	G	6
Pearlitic malleable iron, ASTM A 220 – 55 T, grade 60003	cold - melt, air - furnace; reheat, quench and temper	60003 PMI—(L)	H	7
	duplex, cupola—electric; air-quench and temper	60003 PMI—(C)	J	8
Pearlitic malleable iron, ASTM A 220 – 55 T, grade 80002	duplex, cupola—air furnace; oil-quench and temper	80002 PMI—(W)	K	9
Nodular iron, ASTM A 339 – 55, grade 80-60-03	as-cast; cupola	80-60-03 N.I.	L	10
Nodular iron, ASTM A 339 – 55, grade 60-45-10	cupola; full anneal	60-45-10 N.I.	M	11
Mild steel, AISI 1020	¼ in. rolled plate	1020 steel	N	12
Low-alloy steel	¼ in. rolled plate	Corten steel	P	13
Copper-containing carbon steel	¼ in. rolled plate	Cu-bearing steel	S	14

Of each of the 12 cast metals, 120 plates were procured, cast to the 4 by 6 by $\frac{1}{2}$-in. size. Half (that is, 60) of each of these lots were machined on both 4 by 6 in. surfaces, approximately $\frac{1}{8}$ in. being removed from each surface, leaving a plate $\frac{1}{4}$ in. thick. Of the three steels, 60 plates of each type were procured in the 4 by 6 by $\frac{1}{4}$-in. size and no machining was done thereon. The edges of none of the specimens were machined.

The chemical composition of each of

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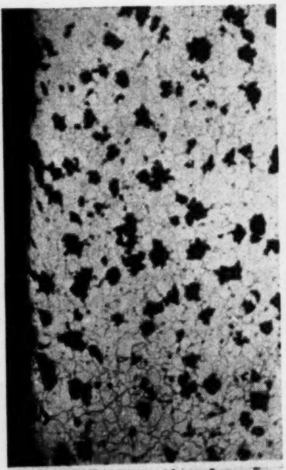
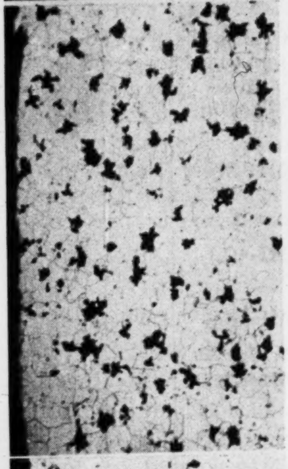
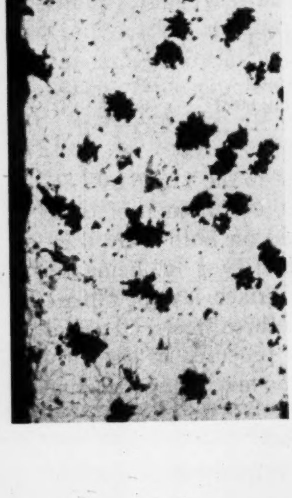
[3] Standard Specification for Nodular Iron Castings (A 339 – 55), 1958 Book of ASTM Standards, Part 1, p. 1458.



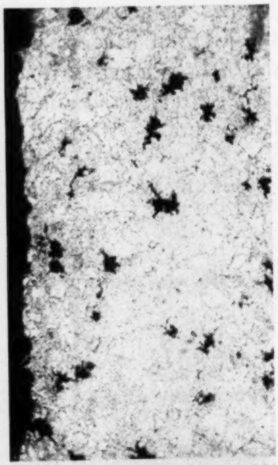
Malleable Plus Copper



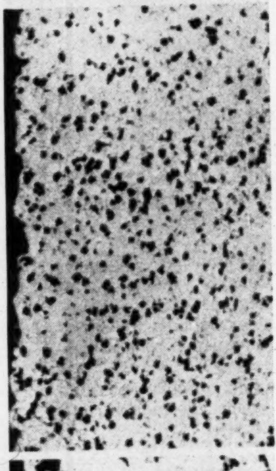
35018 Malleable



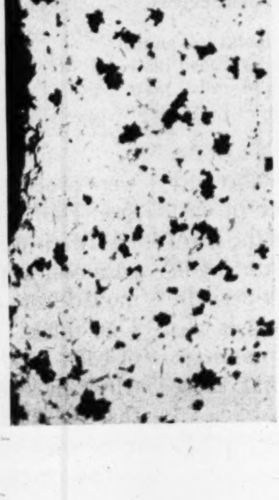
35018 Malleable



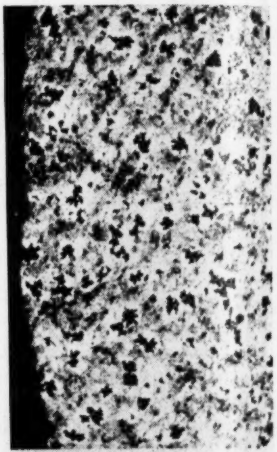
45010 Pearlitic Malleable



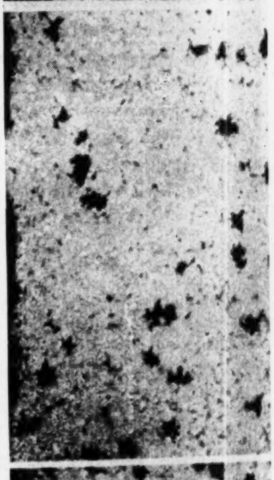
32510 Malleable, High Silicon



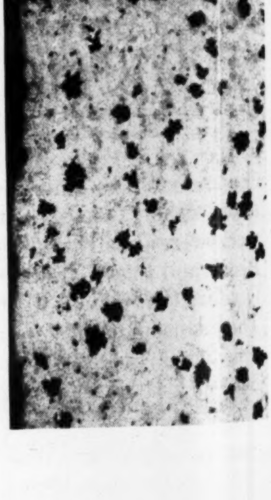
32510 Malleable, Medium Silicon



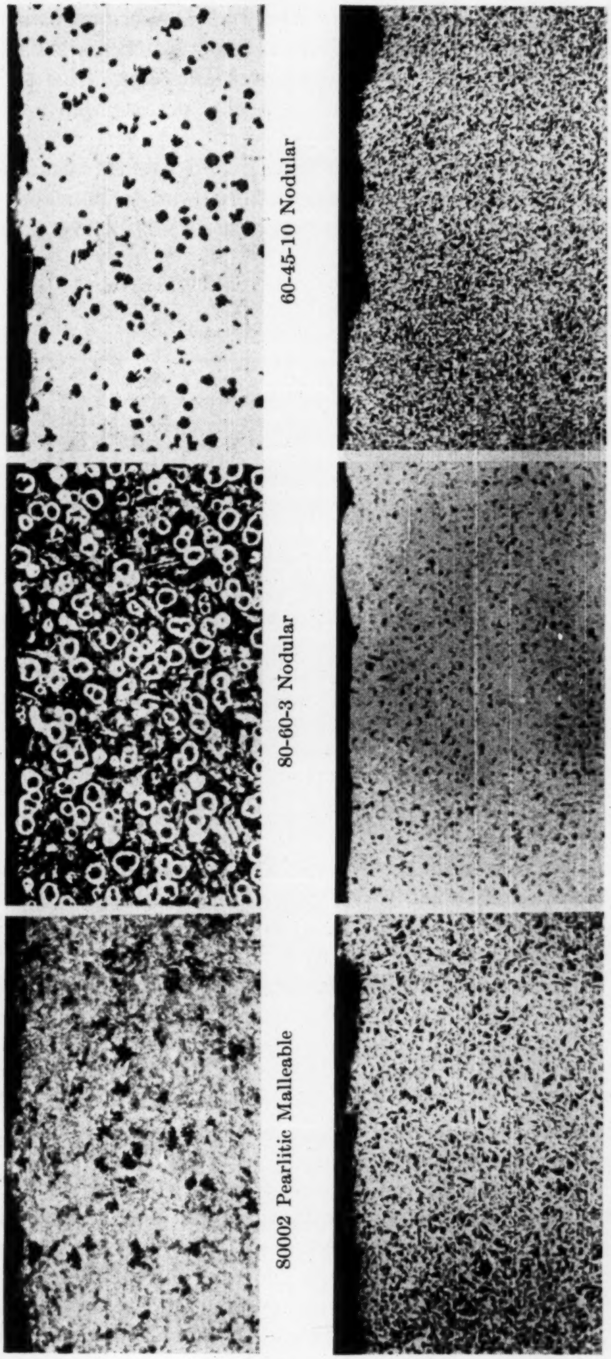
60003 Pearlitic Malleable



60003 Pearlitic Malleable



45010 Pearlitic Malleable, Manganese



80002 Pearlitic Malleable

80-60-3 Nodular

60-45-10 Nodular

1020 Rolled Steel

Corten Steel

Copper Bearing Steel

FIG. 1.—Photomicrographs of Basis Metals; Edge of Specimen at Top. Nital Etch (×40 approx).

the 15 metals is given in Table II. These represent actual analyses by the same laboratory of each of the materials in the condition in which they were corrosion-tested. Because of possible sampling difficulties through loss of the graphitic carbon, total carbon determinations were not run on the malleable irons. For guidance, the total carbon content of the white irons from which the three 35018 standard malleable irons and the 45010 and 60003-(L) pearlitic malleable irons would normally be in the range 2.20 to 2.40 per cent carbon. The other sets of malleable and pearlitic malleable irons would be 2.40 to 2.60 per cent total carbon.

TABLE II.—COMPOSITION OF TEST MATERIALS.

Material	Element, per cent								
	Carbon	Silicon	Manganese	Sulfur	Phosphorus	Chromium	Nickel	Molybdenum	Copper
35018 malleable iron—(L)	[a]	1.12	0.32	0.075	0.155	0.01	nil	nil	0.08
35018 malleable iron—(E)	[a]	1.11	0.36	0.070	0.140	0.01	nil	nil	0.10
35018 malleable iron with copper	[a]	1.12	0.36	0.095	0.150	0.01	0.01	nil	0.65
32510 malleable iron—(N)	[a]	1.11	0.57	0.214	0.054	0.02	0.01	nil	0.14
32510 malleable iron—(A)	[a]	1.60	0.45	0.156	0.037	0.02	0.01	nil	0.08
45010 pearlitic M.I.—(L)	[a]	1.14	0.36	0.072	0.153	0.01	0.01	nil	0.06
45010 pearlitic M.I., Mn	[a]	1.16	0.87	0.086	0.145	0.03	0.02	nil	0.10
60003 pearlitic M.I.—(L)	[a]	1.12	0.35	0.070	0.155	0.02	nil	nil	0.07
60003 pearlitic M.I.—(C)	[a]	1.31	0.45	0.115	0.054	0.02	0.05	nil	0.05
60-45-10 nodular iron	3.57	2.32	0.30	0.018	0.025	0.03	0.08	0.05	0.07
80-60-03 nodular iron	3.70	2.37	0.28	0.023	0.027	0.01	0.08	0.04	0.05
Mild steel, AISI 1020	0.18	0.01	0.79	0.030	0.012	0.04	0.03	nil	0.05
Copper-bearing steel	0.18	0.04	0.63	0.045	0.015	0.03	0.01	nil	0.26
Corten steel	0.09	0.42	0.32	0.042	0.120	1.02	0.49	0.07	0.46

[a] Not determined.

NOTE.—All analyses were run on actual test specimens at one laboratory.

Representative microstructures of the 15 different materials are presented in Fig. 1. These were taken just under the surface of specimens exposed 1 yr at State College. Hardness test results, determined on representative specimens in the ¼-in. thickness, are listed in Table III.

IDENTIFICATION OF SPECIMENS

To serve as a locating point the upper left corner of all specimens was ground off at 45 deg. Notches were machined into the bottom (4-in.) edge to identify the material, in accordance with the system noted in the last vertical column of Table I. To confirm identity of the plate material and to indicate precisely the exposure history of every individual plate, a 3-hole numbering system was employed. By marking off ⅜-in. centers laterally as well as longitudinally on a 4 by 6-in. surface, it is possible to locate 10 centers across the 4-in. dimension and 15 centers down the 6-in. dimension. Any one such center could signify a two-digit number. By drilling three ⅛-in. diameter holes at appropriate centers, three two-digit numbers are generated, which suffice to identify each test plate fully. Reading from left to right and top to bottom, the first hole designates the material and year of removal; the second hole indicates the number of the specimen and whether the exposed surface is as-cast or machined. The third hole locates the exposure site, as specified in Fig. 2. For example, plate J3-J8-N8 would be grade 60003 pearlitic malleable iron, obtained from source "C," third exposure period (7 yr), machined, and exposed at State College, Pa. It would have 8 notches on the bottom edge. By this system of both notches and holes, the material from which any plate is made can be determined either by the number of notches or by the position of the first hole.

TABLE III.—HARDNESS OF BASIS METALS.

Material	Hardness, Brinell Hardness Number		
	max	min	avg[a]
35018 malleable iron—(L)	131	131	131
35018 malleable iron—(E)	121	118	119
35018 malleable iron with copper	152	143	146
32510 malleable iron—(N)	116	114	115
32510 malleable iron—(A)	128	126	127
45010 pearlitic malleable iron—(L)	201	197	198
45010 pearlitic malleable iron, Mn	170	170	170
60003 pearlitic malleable iron—(L)	229	229	229
60003 pearlitic malleable iron—(C)	187	187	187
80002 pearlitic malleable iron—(W)	255	255	255
60-45-10 nodular iron	143	143	143
80-60-03 nodular iron	229	207	214
1020 steel, hot-rolled plate	137	137	137
Copper-bearing steel	131	131	131
Corten (low-alloy) steel	156	156	156

[a] Average of three readings.

NOTE.—Hardness determinations made on ¼ in. thick specimens.

CLEANING AND WEIGHING

All castings and rolled plates were thoroughly cleaned to remove possible traces of rust, scale, and cutting oils. They were then weighed to 2-mg accuracy on a laboratory balance of 2000-g capacity. Following weighing, each specimen was sealed in an individual envelope that had been treated with a proprietary volatile corrosion inhibitor. The plates were then crated, shipped to the appropriate test site, and stored under cover until mounted.

TEST SITES AND MOUNTING OF SPECIMENS

Two marine sites (Kure Beach, N. C. and Point Reyes, Calif.), two industrial sites (East Chicago, Ind., and Newark, N. J.), and one rural site (State College, Pa.) were finally selected. The site at Kure Beach was the one 80 ft from the ocean. Originally one of the locations

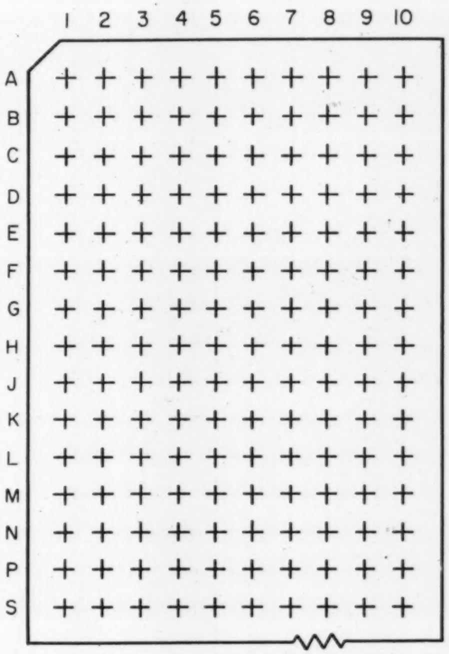


FIG. 2.—Identification of Specimens.

Distance between centerline of ⅛-in. diameter holes = ⅜ in.

Specimen identification: Notches and first hole (letter) (see Table I).

Year of exposure: First hole, numbers 1, 2, 3, or 4.

Specimen number and machined-or-unmachined: second hole (letter and number).

Exposure site: Third hole.

State College, Pa.	N8
Point Reyes, Calif.	S8
Kure Beach, N. C.	S10
Newark, N. J.	P9
East Chicago, Ind.	N10

was to have been near Galveston (below Freeport), Tex., but this was supplanted by State College in order to include a rural site.

All specimens were secured with porcelain insulating knobs to corrosion-resistant metal frames, which are inclined 30 deg from the horizontal and are some 2 to 4 ft from the ground. The exposure is essentially south at East Chicago, Newark, and State College. The Point Reyes site faces west to the Pacific; the Kure Beach site faces east and is parallel to the Atlantic Ocean shore. Figure 3 presents a general view of a typical rack of specimens under exposure. A close-up view of several plates (Fig. 4) shows the details of specimen markings and method of mounting.

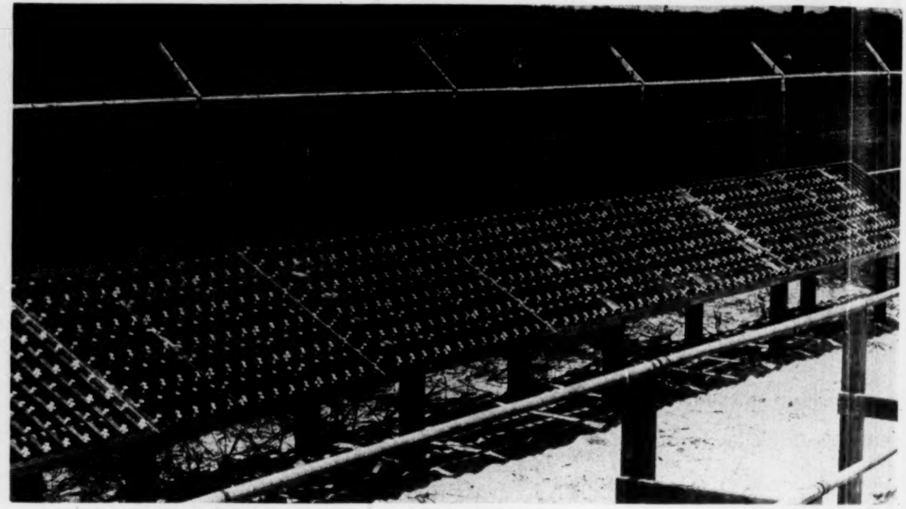


FIG. 3.—General View of a Typical Rack for Exposing 405 Test Specimens at the Start of This Project (Kure Beach, N. C., 80-ft Site).

At each of the five test locations, 324 specimens were mounted, providing 81 plates for each of the four years of removal. All 12 cast metals were exposed with both as-cast and machined surfaces. All sets, 27 in all, for each of the four exposure periods, were in triplicate. No coated specimens were included in this test.

REMOVAL AND CLEANING

After one year of atmospheric exposure, all sites were serviced. The first-year plates were removed, boxed, and shipped to a common point for cleaning. The plates remaining on the racks were examined, broken insulating knobs were replaced, and bolts tightened. All plates were cleaned by the sodium hydride descaling process which removes only the oxides, leaving the base metal unattacked. The cleaned and dried plates were then reweighed and weight losses calculated.

In this investigation no attempt was made to run pit depth measurements because of the time involved and the considerable scatter that has been reported by other investigators (**1**)[4] in making such determinations.

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[4] The boldface numbers in parentheses refer to the list of references appended to this paper.

Results and Discussion

The total weight loss and weight loss per unit surface area for the triplicate specimens in each of the 135 sets were averaged and standard deviations computed from the loss/area data. Weight loss per unit area was determined by dividing the net weight loss (in grams) by 3.74 sq dm for the $\frac{1}{2}$ in. thick plates and by 3.42 sq dm for the $\frac{1}{4}$ in. thick plates. It was felt that this was of sufficient accuracy for a test of this kind, since all plates were reasonably uniform in dimensions. The results are listed in Tables IV, V, and VI.

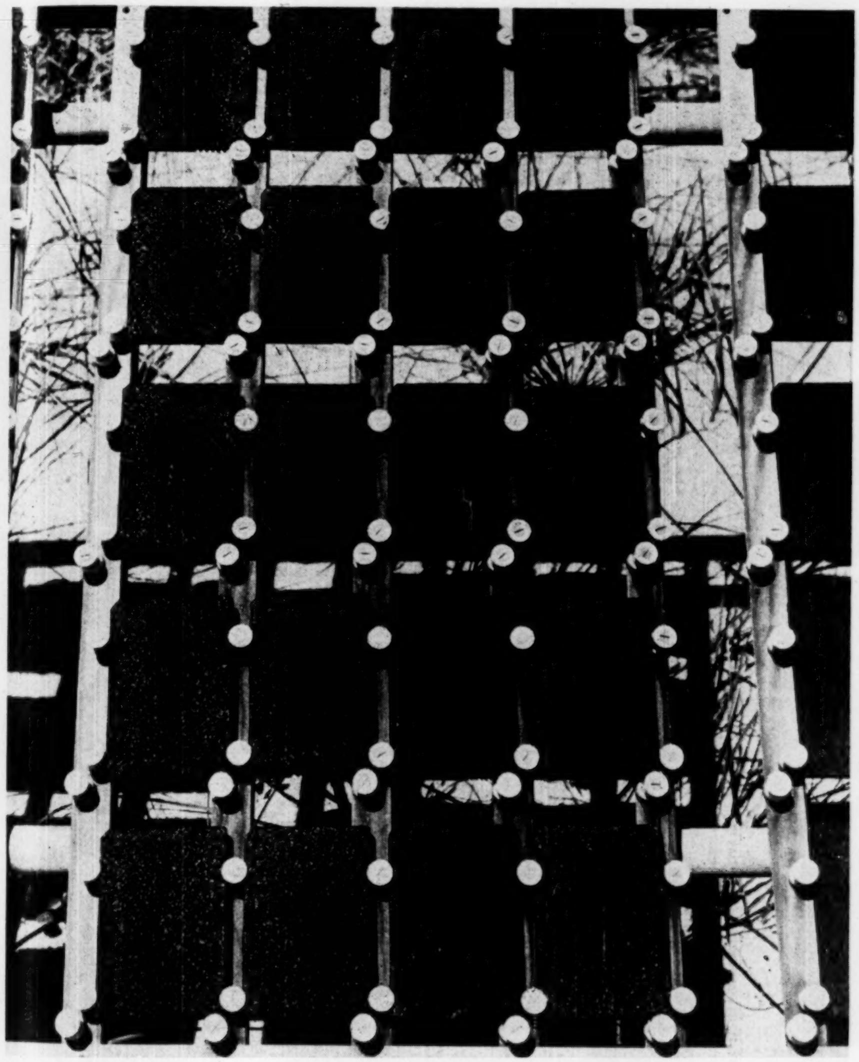

Fig. 4.—Close-up View of a Group of Test Specimens After Exposure to the Atmosphere for 1 yr.

Individual weight losses were found to fall well within the limits attributable to chance. Agreement within each set, that

TABLE IV.—WEIGHT LOSS RESULTS FOR UNMACHINED ½ IN. THICK CAST SPECIMENS.

Type Site	Rural			Marine						Industrial						All Sites
Location	State College, Pa.			Point Reyes, Calif.			Kure Beach, N. C.			Newark, N. J.			East Chicago, Ind.			
Material	Weight Loss, avg[a]		Standard Deviation	Weight Loss, avg[a]		Standard Deviation	Weight Loss, avg[a]		Standard Deviation	Weight Loss, avg[a]		Standard Deviation	Weight Loss, avg[a]		Standard Deviation	Weight Loss, avg
	Net g	g per sq dm		Net, g	g per sq dm		Net, g	g per sq dm		Net, g	g per sq dm		Net, g	g per sq dm		g per sq dm
35018 M.I. (L)	7.77	2.08	0.15	12.35	3.30	0.22	13.47	3.60	0.14	12.28	3.28	0.21	16.46	4.40	0.11	3.33
35018 M.I. (E)	7.46	2.00	0.05	10.28	2.75	0.11	12.69	3.39	0.12	12.85	3.44	0.19	13.81	3.69	0.12	3.05
35018 M.I. + Cu	7.29	1.95	0.07	10.01	2.68	0.20	11.75	3.14	0.14	10.11	2.70	0.06	11.84[b]	3.17[b]	0.06[b]	2.73
32510 M.I. (N)	10.21	2.73	0.06	15.33	4.10	0.10	19.22	5.14	0.20	20.87	5.58	0.17	23.08	6.17	0.43	4.74
32510 M.I. (A)	8.72	2.33	0.04	14.23	3.80	0.19	17.93	4.79	0.11	23.82	6.37	0.36	38.78	10.37	0.31	5.53
45010 PMI (L)	7.30	1.95	0.08	12.30	3.29	0.03	14.16	3.79	0.35	13.48	3.61	0.14	17.36	4.64	0.12	3.46
45010 PMI, Mn	8.76	2.34	0.06	13.75	3.68	0.15	13.77	3.68	0.29	14.52	3.88	0.28	17.73	4.74	0.11	3.67
60003 PMI (L)	7.21	1.93	0.10	12.79	3.42	0.16	13.64	3.65	0.22	13.23	3.54	0.08	15.48	4.14	0.25	3.33
60003 PMI (C)	7.17	1.92	0.05	12.56	3.36	0.10	12.91	3.45	0.08	12.04	3.22	0.14	14.17	3.79	0.06	3.15
80002 PMI (W)	7.39	1.98	0.11	14.37	3.84	0.12	12.87	3.44	0.11	14.70	3.93	0.31	18.10	4.84	0.22	3.61
60-45-10 N.I.	12.26	3.28	0.30	25.60	6.85	0.74	20.66	5.52	0.19	17.56	4.70	0.18	15.21	4.07	0.33	4.88
80-60-03 N.I.	8.50	2.27	0.06	15.47	4.13	0.25	13.14	3.51	0.15	15.63	4.18	0.20	19.48	5.21	0.06	3.86
All cast plates	8.34	...	...	14.09	...	...	14.68	...	...	15.09	...	...	18.46	...	...	...
	...	2.23	...	...	3.77	...	...	3.93	...	...	4.04	...	...	4.93	...	3.78
	...	...	0.09	...	...	0.20	...	...	0.18	...	...	0.19	...	...	0.18	...

[a] Each reported value represents the average for three companion specimens.

[b] Average of only 2 companion plates.

TABLE V.—WEIGHT LOSS RESULTS FOR CAST SPECIMENS MACHINED TO ¼-IN. THICKNESS.

Type Site	Rural			Marine						Industrial						All Sites
Location	State College, Pa.			Point Reyes, Calif.			Kure Beach, N. C.			Newark, N. J.			East Chicago, Ind.			
Material	Weight Loss, avg[a]		Standard Deviation	Weight Loss. avg[a]		Standard Deviation	Weight Loss, avg[a]		Standard Deviation	Weight Loss, avg[a]		Standard Deviation	Weight Loss, avg[a]		Standard Deviation	Weight Loss, avg
	Net, g	g per sq dm		Net, g	g per sq dm		Net, g	g per sq dm		Net, g	g per sq dm		Net, g	g per sq dm		g per sq dm
35018 M.I. (L)	7.75	2.27	0.08	9.58	2.80	0.03	12.93	3.78	0.08	18.77	5.49	0.08	20.81	6.08	0.16	4.08
35018 M.I. (E)	7.02	2.05	0.13	9.29	2.72	0.06	11.56	3.38	0.06	16.04	4.69	0.27	18.34	5.36	0.12	3.64
35018 M.I. + Cu	6.92	2.02	0.03	9.17	2.68	0.08	11.20	3.28	0.10	12.41	3.63	0.03	13.80	4.03	0.11	3.13
32510 M.I. (N)	9.16[b]	2.68[b]	0.04	13.73	4.01	0.08	17.71	5.18	0.25	38.95	11.39	1.21	39.55	11.57	0.43	6.97
32510 M.I. (A)	9.66	2.83	0.15	12.60	3.68	0.08	16.19	4.74	0.25	43.25	12.65	1.00	45.01	13.16	0.43	7.41
45010 PMI (L)	6.51	1.90	0.08	8.01	2.34	0.06	11.19	3.27	0.02	10.53	3.08	0.08	15.23	4.45	0.10	3.01
45010 PMI, Mn	6.53	1.91	0.03	9.49	2.77	0.06	11.91	3.48	0.05	11.20	3.27	0.01	15.28	4.47	0.13	3.18
60003 PMI (L)	6.20	1.81	0.08	8.79	2.57	0.10	10.87	3.18	0.08	10.30	3.01	0.13	14.17	4.14	0.02	2.94
60003 PMI (C)	8.04	2.35	0.06	9.64	2.82	0.15	12.29	3.59	0.07	14.52	4.25	0.20	20.29	5.93	0.15	3.79
80002 PMI (W)	7.26	2.12	0.03	9.43	2.76	0.06	11.55	3.38	0.02	13.56	3.96	0.08	16.48	4.82	0.20	3.41
60-45-10 N.I.	7.00	2.05	0.06	9.17	2.68	0.06	11.22	3.28	0.16	11.00	3.22	0.12	16.86	4.93	0.24	3.23
80-60-03 N.I.	6.18	1.81	0.02	8.51	2.49	0.05	10.24	2.99	0.06	8.79	2.57	0.05	11.64	3.40	0.06	2.65
All cast plates	7.35	...	...	9.78	...	...	12.41	...	...	17.44	...	...	20.62	...	...	...
	...	2.15	...	...	2.86	...	...	3.63	...	...	5.10	...	...	6.03	...	3.95
	...	...	0.07	...	...	0.07	...	...	0.10	...	...	0.27	...	...	0.18	...

[a] Each reported value represents the average for three companion specimens.

[b] Average of only two companion specimens; one plate missing.

TABLE VI.—WEIGHT LOSS RESULTS FOR ROLLED STEEL SPECIMENS, UNMACHINED, ¼ IN.

Type Site	Rural			Marine						Industrial						All Sites
Location	State College, Pa.			Point Reyes, Calif.			Kure Beach, N. C.			Newark, N. J.			East Chicago, Ind.			
Material	Weight Loss, avg[a]		Standard Deviation	Weight Loss, avg[a]		Standard Deviation	Weight Loss, avg[a]		Standard Deviation	Weight Loss, avg[a]		Standard Deviation	Weight Loss, avg[a]		Standard Deviation	Weight Loss, avg
	Net, g	g per sq dm		Net, g	g per sq dm		Net, g	g per sq dm		Net, g	g per sq dm		Net, g	g per sq dm		g per sq dm
1020 steel	12.12	3.54	0.14	30.46	8.91	0.31	37.69	11.02	0.88	21.88	6.40	0.60	18.41	5.38	0.41	7.05
Corten steel	9.09	2.66	0.08	13.26	3.88	0.41	14.97	4.38	0.10	14.72	4.30	0.08	14.34	4.19	0.16	3.88
Copper-bearing steel	11.53	3.37	0.10	18.47	5.40	0.17	26.81	7.84	0.20	19.04	5.57	0.13	16.64	4.87	0.10	5.41
All steels	10.91	...	...	20.73	...	...	26.49	...	...	18.54	...	...	16.46	...	...	...
	...	3.19	...	...	6.06	...	...	7.75	...	...	5.42	...	...	4.81	...	5.45
	...	...	0.11	...	...	0.30	...	...	0.39	...	...	0.27	...	...	0.22	...

[a] Each reported value represents the average of three companion specimens.

is, on replicate specimens, was generally very good.

Summary results are presented in Fig. 5 in the form of three bar graphs. In each group the bars are arranged in the order of increased attack from left to right.

As-Cast Versus Machined Surfaces:

Based on grand averages, the machined plates lost more weight than the specimens exposed with as-cast surfaces. However, when the individual materials are examined, the pattern tended to be somewhat erratic, with 7 metals showing greater corrosion rates for the machined specimens and 5 showing less attack in the machined condition. The two 32510 malleable irons as machined lost 47 and 34 per cent more than the unmachined, whereas the two machined nodular irons exhibited 34 and 31 per cent less weight loss than the unmachined.

Microstructural studies failed to reveal any specific cause for such anomalies. Visual observations of the plates after 1 yr exposure, but before cleaning, revealed large flakes of a bluish scale on the 60-45-10 nodular iron unmachined surfaces which could perhaps be related to the atmosphere of the heat-treating furnace. Possibly this scale contributed to increased attack on the as-cast surfaces. Heavy scaling was also observed on the two 32510 malleable irons. This condition seemed worse on the machined specimens than on the unmachined. Therefore, some correlation appears to exist between visual appearance and actual weight loss. Other investigations have found this to be the case, even to the extent of finding a correlation between color of scale and weight loss (3).

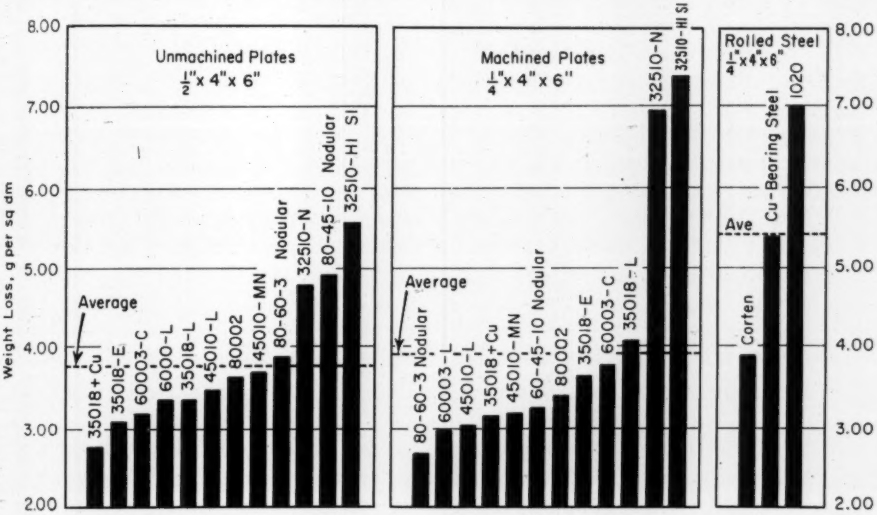

FIG. 5.—Average Weight Loss of Specimens Exposed 1 yr, All Sites.

The bar graphs of Fig. 5 reveal a noticeably higher attack for the two 32510 malleable irons than for the other cast metals, particularly in the machined condition. No acceptable explanation can be offered at this time on the basis of metal history, chemistry, or structure. While both of these sets were produced by two large completely unrelated foundries, it should not be construed to suggest that all malleable irons of grade 32510 would be attacked as severely as were those employed in this project. The 60003-(C) and 80002-(W) pearlitic malleable irons were made from 32510-base iron, but corrosion rates for these were normal.

Comparison of Materials:

On the basis of grand averages, the two nodular irons showed 21 per cent greater attack than did the ten malleable and pearlitic malleable irons as a group in the unmachined condition. However the reverse was true for companion material exposed in the machined state, namely, 2.94 g per sq dm for nodular irons as against 4.16 g per sq dm for all malleable irons combined. Were the two abnormal 32510 malleable irons excluded from these averages, the grand average for the eight malleable irons, machined, would be 3.40 g per sq dm—roughly the same as the corrosion rates for nodular irons as machined.

There seemed to be no significant difference in resistance to atmospheric corrosion between ferritic (standard) and pearlitic malleable irons. If the unusually high results for the two 32510 malleable irons were neglected, the averaged values for both groups are almost identical (namely 3.33 versus 3.35 g per sq dm, respectively) combining machined and unmachined test results. Specimens exposed with as-cast (unmachined) surfaces showed somewhat greater corrosion in the pearlitic malleable irons as a group than in the standard malleable materials. On the other hand, in the machined condition the standard malleable irons exhibited slightly higher weight loss than did the pearlitic malleable irons.

With regard to the nodular irons tested, the ferritic material (grade 60-45-10) was attacked more than the pearlitic (grade 80-60-3) in both machined and unmachined conditions (by 22 and 26.5 per cent, respectively). Since there was only one sample lot of each of the two nodular iron grades, these differences could be peculiar to the particular stock employed for the test. In other words, all nodular irons might not necessarily follow the same pattern.

Benefits of Alloying:

The loss in weight of unalloyed 1020 steel was 87 and 78 per cent greater than the averages for all cast specimens unmachined and machined respectively. Increasing the copper content up to about the 0.25 per cent level reduced the loss in weight from 7.05 g per sq dm to 5.41 g per sq dm (average for all sites), or some 23 per cent. Nevertheless, even with the added copper, the copper-bearing steel underwent some 40 per cent greater corrosive attack than the average of all cast specimens. Additional alloying of the steel (that is, "Corten") improved further its resistance to atmospheric attack, so that the weight loss was roughly equivalent to the average for all the unalloyed cast metals. Other studies (2) have demonstrated that alloying with copper, phosphorus, nickel, or chromium is beneficial, and this is borne out here.

The advantage of the 0.25 per cent copper in steel was most pronounced at the marine locations, where 29 and 39.5 per cent (Kure Beach and Point Reyes, respectively) reduction in weight loss was accomplished. The least benefit, or 5 per cent, occurred at the rural (State College) site. The industrial locations gave intermediate benefits, namely, 9.5 and 13 per cent at East Chicago and Newark, respectively.

In malleable irons the addition of about 0.5 per cent copper was advantageous, primarily in industrial atmospheres where an average weight loss reduction of 22 per cent resulted. In marine atmospheres this reduction was only about 3.5 per cent, while in rural environments a mere 2 per cent reduction was realized. This is qualitatively in accord with the findings of other investigators (3), who have reported that copper is most beneficial in sulfurous industrial atmospheres and that the effect of copper is greater in steels than in irons (4). Over exposure periods up to $3\frac{1}{2}$ yr, 0.1 per cent copper has been reported (2) to suffice in improving corrosion resistance, with little benefit being derived from greater amounts. However, over longer periods (that is, 15 yr) continued improvement has been obtained from higher copper levels (up to 1.0 per cent). As the present project continues over the longer term, such trends will be evaluated for the materials included in this work.

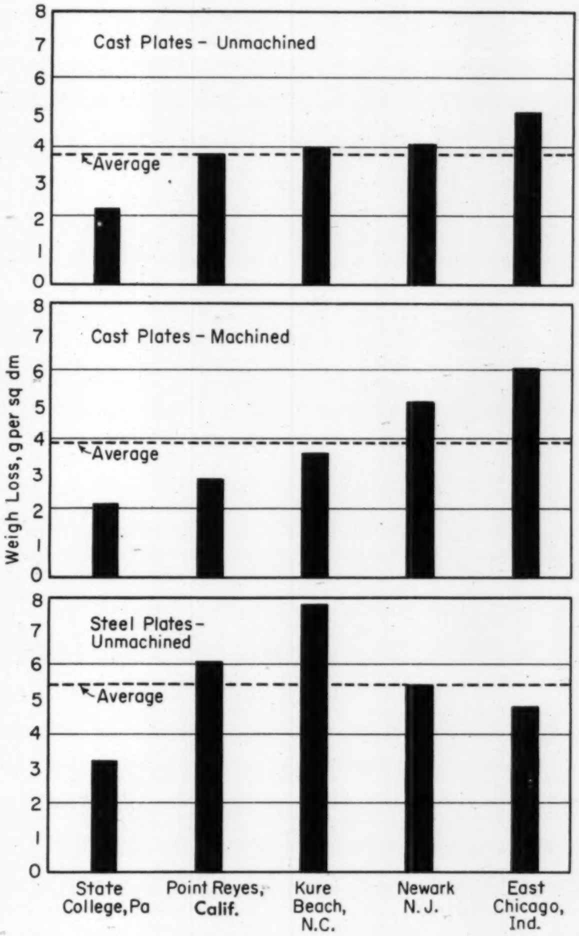

FIG. 6.—Average Weight Losses for All Specimens at Each of the Five Exposure Sites.

Effect of Environment:

References have been made in the discussion above to how the various metals were affected differently by the atmosphere at the several sites. In Fig. 6 the averaged results for all materials at the five test locations are graphically pictured. It is very apparent that the mildest environment prevailed at the rural (State College) site. For the cast alloys, the most severe atmospheres proved to be at the industrial sites, East Chicago being decidedly worse than Newark. Intermediate corrosive attack on the cast alloys occurred at the marine locations. For the steels, on the other hand, the marine locations were the most severe, while the industrial sites were intermediate. For all materials exposed at marine sites, Kure Beach (80-ft site) was more harsh than Point Reyes. It had previously been discovered that environmental conditions at the Kure Beach 80-ft site are more severe on unalloyed steels than at all other sites studied (3,5,6). A comparison of the results of this investigation with the findings of others for mild steel is presented in Table VII. The ranking of sites falls in satisfactory order, although some disagreement exists in specific values for some of the sites. This could be attributable to differences in condition of the basis steel, test procedures, abnormal weather for certain years, season of year for the initial exposure, and physical changes in the vicinity of the site that may influence the corrosiveness of the atmosphere over the years. It is suspected (3) that eventual corrosion is strongly affected by whether the weather is wet or dry at the time of the initial exposure. While a single 1-yr test should suffice to indicate relative corrosivity in rural and mild industrial locations, at least three successive yearly exposures have been suggested (6) before a reliable corrosivity index can be obtained for steel in severe industrial or marine atmospheres.

TABLE VII.—CORROSION OF UNALLOYED ROLLED STEEL EXPOSED 1 YR.

Attack	Site	Average Weight Loss, g			
		Present Study, 4 by 6 by ¼-in. Plates	LaQue (3)		Larrabee and Ellis (6), 4 by 6 in. Plates
			2 by 4-in. Plates	Approximate Conversion to 4 by 6 in.	
Severe	Kure Beach, N. C., 80-ft site	37.7	70.5	211.5	90.2
	Daytona Beach, Fla. (marine)	...	20.4	61.2	48.5
	Point Reyes, Calif.	30.5	...	...	12.2
	Frodingham, British Isles	...	14.8	44.4	...
	New York, N. Y.; fall installation	...	...	...	40.5
Moderate	Newark, N. J.	21.9	...	...	...
	New York, N. Y.; spring installation	...	...	...	21.1
	Kure Beach, N. C., 800-ft site	...	5.8	17.4	13.3
	Kearny, N. J.	...	7.8	23.4	20.4
	East Chicago, Ind.	18.4	...	...	...
Mild	State College, Pa.	12.1	3.8	11.4	6.8
	South Bend, Pa.	...	...	...	10.5
	Middletown, Ohio	...	...	...	8.4
	Vancouver (Esquimalt), B. C.	...	...	...	3.3

The bar graph of Fig. 7 illustrates the effect of environment on five specific materials after 1 yr exposure. Malleable iron No. 35018-E and malleable iron containing copper, No. 35018 + Cu, cast from the same heat as the 35018-E specimens, are compared with the three rolled steels which vary from no alloy to some 2 per cent total alloy. From the bar graph the beneficial effect of copper in malleable iron in industrial atmospheres is quite apparent. The advantage of low copper surface offers some protective qualities to malleable iron in industrial atmospheres, regardless of whether alloyed with copper or not.

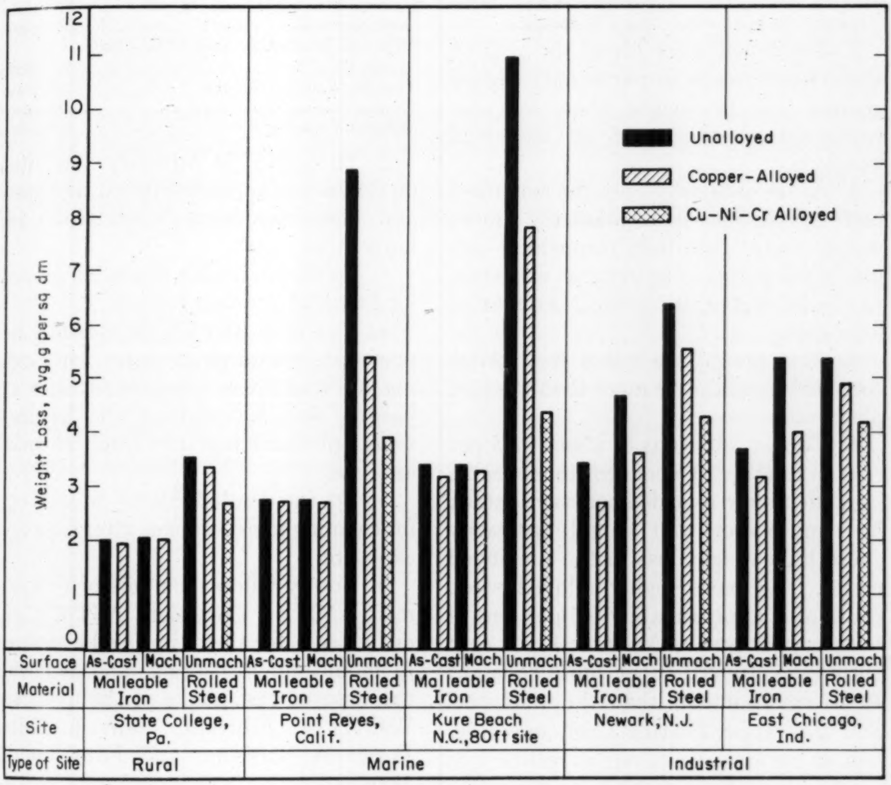

Fig. 7.—Effect of Environment on Five Materials After 1-yr Exposure.

additions to mild steel is very noticeable, especially for service in marine environments. While the steel alloyed with copper, chromium, and nickel provided better resistance to attack at all sites than did the low copper-bearing steel, it still was not as good as the copper-bearing malleable iron at any site, nor as good as the unalloyed malleable iron at rural and marine locations. The severe attack of marine atmospheres on mild steel is very marked in the bar graph. Also, there is evidence that the as-cast (unmachined)

Conclusions

Before summarizing the findings of this report, it would be well to emphasize that whatever conclusions are drawn must be based on only the one exposure period. It is quite possible that the degree of corrosive attack and the ranking of materials could be quite different for longer or shorter periods than one year.

1. For all materials, corrosive attack at State College (rural) was consistently less than at industrial and marine sites.

2. For cast specimens, regardless of whether machined or unmachined, the atmospheres at the two industrial sites proved to be more severely corrosive than at the rural or marine locations, East Chicago being worse than Newark.

3. For rolled steel plate, the marine sites (Kure Beach in particular) caused greater loss in weight than was encountered at the rural or industrial locations.

4. At the industrial sites, the machined cast specimens lost markedly more weight than did their companion unmachined plates. The reverse was true, to a lesser degree, at the rural and marine locations.

5. As a group, the rolled steel plates corroded significantly more than the cast plates collectively.

6. Copper additions of about 0.25 per cent to steel and 0.5 per cent to malleable iron effectively retarded corrosive attack in atmospheres that would ordinarily cause high weight loss in the unalloyed metal. The more highly alloyed steel, containing approximately 0.5 per cent of both copper and nickel and 1 per cent chromium, proved to be more resistant to all environments than the mild steel and the copper-bearing steel; nevertheless, in general, it was not as resistant to environmental corrosion as were the majority of unalloyed cast malleable and nodular irons.

Acknowledgments:

The author wishes to express his appreciation to the following individuals and organizations for assistances in carrying out this program:

1. To the firms who donated the specimen stock for this project:

Albion Malleable Iron Co.	120 plates
Central Foundry Division, General Motors Corp.	120 plates
Eastern Malleable Iron Co.	360 plates
Link-Belt Co.	360 plates
National Malleable and Steel Castings Co.	120 plates
Textile Machine Works	240 plates
United States Steel Corp.	180 plates
Wagner Castings Co.	120 plates

2. To the ASTM Advisory Committee on Corrosion for providing test rack space and mounting frames, insulators, and bolts at all sites.

3. To the Malleable Founders' Society for financial assistance.

4. To Chain Belt Co. for cleaning and packaging the original plates, for weighing all specimens before and after exposure, and for making the hardness tests, photomicrographs and chemical analyses.

5. To the United States Steel Corp. for cleaning the specimens after their 1-yr exposure.

6. To my fellow members of Subcommittee VII on Corrosion, ASTM Committee A-7, for their time and assistance in mounting, inspecting, and removing the exposed test plates at the five sites: Walter M. Albrecht, Hans J. Heine, James H. Lansing, and Lauriston C. Marshall.

7. To the Eastern Malleable Iron Co., Research Department, for making computations and compiling data for this report.

Respectfully submitted on behalf of the subcommittee,

GORDON B. MANNWEILER,
Chairman.

REFERENCES

(1) H. R. Copson, "Atmospheric Corrosion of Low Alloy Steels," *Proceedings,* Am. Soc. Testing Mats., Vol. 52, p. 1005 (1952).

(2) H. R. Copson, "Long-Term Atmospheric Corrosion Tests on Low-Alloy Steels," *Proceedings,* Am. Soc. Testing Mats., Vol. 60, p. 650 (1960).

(3) F. L. LaQue, "Corrosion Testing," *Proceed-*

ings, Am. Soc. Testing Mats., Vol. 51, p. 495 (1951).

(4) V. V. Kendall and E. S. Taylerson, "A Critical Study of the ASTM Corrosion Data on Uncoated Commercial Iron and Steel Sheets," *Proceedings,* Am. Soc. Testing Mats., Vol. 29, p. 204 (1929).

(5) J. C. Hudson, "Present Position of the Corrosion Committee's Field Tests on Atmospheric Corrosion," *Journal,* British Iron and Steel Institute, Vol. 148, p. 161 (1943).

(6) C. P. Larrabee and O. B. Ellis, "On Corrosiveness of Atmospheric Test Sites," *Proceedings,* Am. Soc. Testing Mats., Vol. 59, pp. 183–201 (1959).

REPORT OF COMMITTEE A-9
ON
FERRO-ALLOYS*

Committee A-9 on Ferro-Alloys held one meeting during the year: on June 27, 1960, during the Annual Meeting of the Society in Atlantic City, N. J.

Consideration is currently being given by the committee to the preparation of new specifications for ferrocolumbium and silico manganese.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for:

Ferromanganese (**A 99–57 T**),[1] and
Ferromolybdenum (**A 132–59 T**).[1]

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for more than six years without revision:

Standard Specifications for:

Spiegeleisen (**A 98–50**),[1]
Ferrosilicon (**A 100–50**),[1]
Ferrochromium (**A 101–50**),[1]
Ferrovanadium (**A 102–50**),[1]
Ferrotungsten (**A 144–50**),[1]
Molybdenum Salts and Compounds (**A 146–52**),[1]
Ferroboron (**A 323–52**),[1] and
Ferrotitanium (**A 324–52**).[1]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

This report has been submitted to letter ballot of the committee, which consists of 25 members; 22 members returned their ballots, of whom 21 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

S. W. POOLE,
Chairman.

W. H. MAYO,
Secretary.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] 1958 Book of ASTM Standards, Part 1.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE A-10

ON

IRON-CHROMIUM, IRON-CHROMIUM-NICKEL AND RELATED ALLOYS*

Committee A-10 and most of its subcommittees held two meetings during 1960: on June 27 and 28 during the Annual Meeting of the Society in Atlantic City, N. J., and on Jan. 31 and Feb. 1, 1961, during ASTM Committee Week in Cincinnati, Ohio.

The committee has a total membership of 119, of whom 54 are classified as producers, 47 as consumers, and 18 as general interest members.

Unanimous approval has been given to the proposal that Committee E-4 on Metallography be informed that all grain-size requirements in Committee A-10 specifications will refer to Methods E 112 - 60 T, and that the other older grain-size methods will be withdrawn.

On the recommendation of Subcommittee IV, unanimous approval has been given to the publication of the report survey of architectural applications of stainless steels as appended hereto.[1]

NEW TENTATIVES

The committee, jointly with Committee A-1 on Steel, recommends for publication as tentative the following specifications which are appended to the Annual Report of Committee A-1:[2]

New Tentative Specification for:

Centrifugally Cast Austenitic Steel Pipe for High-Temperature Service,

Austenitic Centrifugally Cold-Wrought Pip for High-Temperature Service, and

High-Strength, High-Temperature Bolting Materials with Expansion Coefficients Comparable to Austenitic Steels.

In addition, the committee recommends for publication as tentative the following specifications as appended hereto:[2]

New Tentative Specifications for:

Hot-Cold Rolled Alloy Plate, Sheet and Strip for High Strength at Elevated Temperatures, and

Hot-Cold Worked Alloy Bars for High Strength at Elevated Temperatures.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Specification for Seamless and Welded Austenitic Stainless Steel Pipe (A 312 - 60 T)[3] (Jointly with Committee A-1 on Steel):

Section 8(b).—After the words "chemical analysis" add the phrase "except for spectrochemical analysis."

Tentative Specification for Corrosion-Resisting Chromium Steel Clad Plate, Sheet, and Strip (A 263 - 59 T):[4]

Section 5(b).—Change the first sentence to read as follows:

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] See p. 188.

[2] The new tentatives appear in the 1961 Book of ASTM Standards, Part 1.

[3] 1960 Supplement to Book of ASTM Standards, Part 1.

[4] 1958 Book of ASTM Standards, Part 1.

The alloy cladding metal specified shall conform to the requirements as to chemical composition prescribed for the respective chromium steel in the Specification for Corrosion-Resisting Chromium and Chromium-Nickel Steel Plate, Sheet, and Strip for Fusion-Welded Unfired Pressure Vessels (ASTM Designation: A 240).

Table I.—Delete this table, renumbering the subsequent tables accordingly.

Tentative Specification for Corrosion-Resisting Chromium-Nickel Steel Clad Plate, Sheet, and Strip (A 264 - 59 T):[5]

Section 5(b).—Make the same change as indicated above in Section 5(*b*) of Specification A 263.

Table I.—Delete this table, renumbering subsequent tables accordingly.

Tentative Specification for Corrosion-Resistant Iron-Chromium and Iron-Chromium-Nickel Alloy Castings for General Application (A 296 - 60 T):[3,4a]

Table I.—List new grade CN-7M with the heat treatment requirement to be as agreed upon between the manufacturer and the purchaser.

Table II.—Add a new grade CN-7M with the following chemical requirements:

Carbon, per cent	0.07 max
Manganese, max, per cent	1.50
Silicon, max, per cent	1.50
Sulfur, max, per cent	0.040
Phosphorus, max, per cent	0.040
Chromium, per cent	19.0 to 22.0
Nickel, per cent	27.5 to 30.5
Molybdenum, per cent	2.0 to 3.0
Copper, per cent	3.0 to 4.0

Table III.—Add a new grade CN-7M with the following tensile requirements:

Tensile strength, min, psi	62 500
Yield point, min, psi	25 000
Elongation in 2 in., min, per cent	35

Revisions of Standards, Immediate Adoption

The committee, jointly with Committee A-1 on Steel, recommends for immediate adoption revisions as set forth in the Annual Report of Committee A-1 of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Specifications for:

Seamless and Welded Ferritic Stainless Steel Tubing for General Service (**A 268 - 60**),

Seamless and Welded Austenitic Stainless Steel Tubing for General Service (**A 269 - 60**),

Seamless and Welded Austenitic Stainless Steel Sanitary Tubing (**A 270 - 60**), and

Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (**A 271 - 60**).

Reapproval of Standards

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Standard Specification for:

Chromium-Nickel-Iron Castings (25-12 Class) for High-Temperature Service (**A 447 - 50**).[6]

Standard Recommended Practice for:

Conducting Plant Corrosion Tests (**A 224 - 46**).

Tentatives Continued Without Revision

The committee recommends the continuation without revision of the following tentatives:

Tentative Recommended Practice for:

Boiling Nitric Acid Test for Corrosion-Resisting Steels (**A 262 - 55 T**), and

Conducting Acidified Copper Sulfate Test for Intergranular Attack in Austenitic Stainless Steel (**A 393 - 55 T**).

Editorial Change

The committee recommends editorial change as follows in the Tentative Specification for Nickel and Nickel-Base Alloy Clad Steel Plate (**A 265 - 43 T**):[4]

[4a] Publication of the revision was withheld pending the approval by confirming letter ballot vote in the committee.

[5] 1959 Supplement to Book of ASTM Standards, Part 1.

[6] This standard was formerly designated B 190.

Section 5(b).—Change the first sentence to read as follows:

> The nickel or nickel-base alloy cladding metal specified shall conform to the requirements as to chemical composition prescribed for the respective metal in the Specifications for Nickel Plate, Sheet, and Strip (ASTM Designation B 162); for Nickel-Copper Alloy Plate, Sheet, and Strip (ASTM Designation: B 127); or for Nickel-Chromium-Iron Alloy Plate, Sheet, and Strip (ASTM Designation: B 168).

Table I.—Delete this table and renumber the subsequent tables accordingly.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[7]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II, Editorial (A. G. Cook, chairman) has been particularly active in making effective the editorial surveillance of all Committee A-10 specifications as they come up for committee action. This has resulted in a noticeable improvement in these recommendations. The subcommittee has formulated this editorial policy in its "Editorial Procedure and Form" which has been submitted for formal approval by Committee A-10.

Subcommittee IV on Methods of Corrosion Testing (O. B. Ellis, chairman) has recommended that Recommended Practices A 262, A 393, and A 224 be continued without change.

The task group investigating methods for intergranular corrosion tests reported that agreement between laboratories had been obtained on the ferric sulfate - sulfuric acid tests. Work will now be initiated on heat-treated steels by the five or more participating laboratories. A further report on the progress of this work should be ready by the June, 1961, meeting.

A task group has inspected stainless steel architectural installations including monumental and industrial buildings in Philadelphia, New York, Chicago, Pittsburgh, Dravosburg (Pa.), Cleveland, Miami Beach, and Indian River (Fla). The report of this task group is appended.[1]

The atmospheric exposure of stainless steel sheet, bar, and wire specimens was started three years ago. The first removals will be made during the coming summer and the specimens will be returned to the supplying companies for test.

Sheet specimens representative of several chromium, chromium-nickel, and chromium - nickel - manganese stainless steels were exposed at Newark and Pittsburgh in 1953. These sheets will be inspected in April, 1961, after an exposure of eight years.

Subcommittee V on Mechanical Tests (R. H. Heyer, chairman) currently has a task group engaged in the study of tension testing on thin sheets, many specimens of which have been distributed for interlaboratory tests.

The task group on hardness conversions has made its initial report. It has been decided to extend the scope of activities of this group to additional hardness conversions on other products.

Subcommittee VIII on Specifications for Wrought Products (R. B. Gunia, chairman) has prepared a proposed specification for stainless steel forgings and submitted it for approval. In addition, a task group is studying supplementary nuclear requirements for this specification.

The task group working on stainless steel wire specifications has decided to cover such end uses as cold-heading, weaving, and wire rope. The group also recommends the addition of stainless grades to the spring-wire specification (A 313).

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[7] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

The subcommittee has entered into cooperative effort with the Industrial Fastener Inst. in the preparation of specifications for stainless materials in this field.

The American Standards Assn. has requested consideration of the addition of certain stainless grades to Specification A 276. The objective is to provide industry specifications that can be adopted by Federal agencies. A task group will be appointed to accomplish this objective on as broad a basis as possible.

In answer to a query from The American Society of Mechanical Engineers, the subcommittee has stated that the materials specified in Specification A 276 are similar to those in Specification A 240, and hence, the materials in A 276 having counterparts in A 240 should be acceptable for code construction.

Subcommittee IX on Specifications for Flat Products (G. W. Hinkle, chairman) has recommended the modification of the mechanical properties of types 201 and 202 stainless steel in Specification A 412 and has prepared new General Requirement Specifications for Flat Products. Additionally, modification of the chemical ranges have been made in Specifications A 263, A 264, and A 265. Also, as the result of recommendations from Subcommittee XIII, nuclear supplementary requirements are to be added to Specifications A 240.

Subcommittee X on Specifications for Castings (H. C. Templeton, chairman) has taken over the membership and activities of Subcommittee II on Wrought and Cast Alloys for Structural Use at High Temperatures of Committee B-4 on Electrical Heating and Resistance Alloys, including supervision of Specifications B 190 and B 297, which it recommends be changed to "A" designations. The subcommittee has added a new grade CN-7M to Specification A 296. In addition, cooperating with Subcommittee IX on Steel Tubing and Pipe of Committee A-1, two new specifications have been written to cover austenitic centrifugally cast cold-wrought pipe and centrifugally cast austenitic steel pipe, both for high-temperature service, as noted earlier in this report.

Subcommittee XI on Specifications for Tubular Products (J. J. B. Rutherford, chairman) has, in collaboration with Committee A-1, recommended changes in a number of the tubing specifications under A-1 and A-10 jurisdiction, for the most part for the purpose of clarifying these specifications with respect to newer methods of manufacture. The subcommittee has also been studying the tensile properties of types 304L and 316L with particular respect to boiler code applications; it has a task group working on the preferred silicon content for welding; and is also studying out-of-roundness considerations in tubular products.

Subcommittee XII on Specifications for High-Temperature Super-Strength Alloys (W. F. Simmons, chairman) has completed the work on specifications covering materials in three categories of super-strength alloys: hot-cold worked alloys, precipitation-hardened alloys, and cast alloys. Two new specifications for hot-cold worked alloys are appended to the report.

In joint effort with Subcommittee XXII of Committee A-1, a specification for high-strength bolting material has been prepared as noted earlier in this report.

During the year a revision has been made of *ASTM STP 170A*, Compilation of Chemical Compositions and

Rupture Strengths of Super-Strength Alloys. This publication, available as *STP 170B*, now embraces 163 domestic and 114 foreign alloys.

Subcommittee XIII on Specifications for Nuclear Reactor Structural Materials (M. A. Cordovi, chairman) has prepared nuclear requirements stipulations to be used as supplementary requirement additions to some of the existing Committee A-10 specifications. These have been turned over to the appropriate product committees; one such recommendation is to be included in the current Subcommittee IX revision of Specification A 240.

This report has been submitted to letter ballot of the committee, which consists of 119 members; 72 members returned their ballots, of whom 69 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. L. WYMAN,
Chairman.

G. R. WOODROW,
Secretary.

EDITORIAL NOTE

On August 3, 1961, the Administrative Committee on Standards, acting for the Society, accepted the following recommendations submitted by Committee A-10:

Revision of Tentative Specification for:

Corrosion-Resisting Chromium and Chromium-Nickel Steel Plate, Sheet, and Strip for Fusion-Welded Unfired Pressure Vessels (A 240 – 58 T).

Revision and Reversion to Tentative of Standard Specifications for:

Corrosion-Resisting Chromium Nickel Steel Plate, Sheet, and Strip (A 167 – 58), and
Corrosion-Resisting Chromium Steel Plate, Sheet, and Strip (A 176 – 54).

The revised tentative specifications appear in the 1961 Book of ASTM Standards, Part 1.

APPENDIX

REPORT ON INSPECTION OF CORROSION-RESISTANT STEELS IN ARCHITECTURAL AND STRUCTURAL APPLICATIONS

The inspection committee was of the unanimous opinion that the inspection should reflect the use of stainless steel in industrial buildings as well as in monumental buildings and commercial, office, or retail establishments. The committee also thought that it would be desirable to inspect buildings using AISI types 202 and 316 stainless steel, the latter near the sea coast. Neither of the two types had been included in the previous inspections.[1] To accomplish these objectives, inspections were made in several localities that were not covered by previous committees, namely, Chicago, Ill., Cleveland, Ohio, and Miami and Indian River, Fla.

The inspections were conducted between October 20 and 28, 1960. Some deletion of structures inspected by previous committees was necessary, the principal deletions being the stainless steel used on the various boardwalk sites in Atlantic City. However it was felt that inspections of stainless steels in Miami Beach would be acceptable substitutes for the Atlantic City sites.

A summary of the observations follows:

Pennsylvania Building, 1500 Chestnut St., Philadelphia, Pa.

This building incorporates a considerable amount of type 302 No. 4 finish stainless steel on the window sections and the entrances. Erection was completed in 1949. The metal around the entrances is wiped nightly and the metal around the windows is cleaned frequently. The metal showed no signs of deterioration. There was a small accumulation of dirt on the outside window ledges which could be easily removed and under which there was no trace of rusting.

Philadelphia Savings Fund Society Building, Philadelphia, Pa.

This building was erected in 1932. A considerable amount of stainless steel was used in various parts of the building. The committee was of the opinion that this building was in the same condition as described by the 1954 inspection group.

The chromium stainless steel (probably type 430) used on the interior of the windows of the sun room on the 53rd floor and the small amount of type 302 used on the exterior of some of the windows remained bright and showed no sign of pitting or corrosion. The type 302 sinks, work surfaces, steam table tops, etc. which are in the kitchen of the executive dining room, 53rd floor, retain the expected bright and shiny surfaces. The type 302 stainless, mainly No. 4 finish used in the interior of the building for railings, escalators, column covers, etc., was in excellent condition.

The exterior facings of the vaults are made of a No. 4 finish chromium stainless steel which the management thought was type 410. This area is wiped every three months with a light lemon oil to prevent finger spotting. There were no indications of any pitting or corrosion.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 197 (1939); Vol. 40, p. 118 (1940); Vol. 46, p. 593 (1946); Vol. 48, p. 137 (1948); Vol. 49, p. 138 (1949); and Vol. 55, p. 160 (1955).

The No. 4 finish type 302, used around the entrance, was in excellent condition and showed no signs of deterioration. These entrances are wiped daily with a cloth dampened with ammonia water and cleaned about once a month with whiting.

The ceiling of the subway entrance leading from the sidewalk into the building basement is made of type 302 stainless. There was a very heavy accumulation of dirt on this surface and from the appearance of the metal it is doubtful if it has ever been cleaned. There was some light staining and discoloration as noted in the previous inspection (1954) but it was the consensus that it had not worsened since the previous inspection.

Chrysler Building, New York, N. Y.

The stainless on this building showed no change from the previous inspection. The exterior first three floors incorporate the use of No. 4 finish type 302 stainless steel which is in good condition with no evidence of corrosive attack. It is cleaned twice yearly.

The surfaces of the type 302 sheet used on the tower, erected in 1929–1930, were examined through windows on the 71st floor. The surface was covered with a black deposit, but there was no evidence of any rusting underneath. There was a small amount of superficial red deposit on the window sills, which was thought to be caused by chlorides.

At the 61st floor level, the type 302 stainless steel on the base of the tower and the gargoyles were still in the same condition as in the previous inspection. The surfaces of the tower were covered with dirt but under the dirt there were practically no indications of pitting. The gargoyles had much less dirt adhering to their surfaces than the tower surfaces, presumably because they were in the open and were subject to better cleaning by the weather. There was no evidence of pitting on the surfaces of the gargoyles.

The ornamental figures on the 31st floor level were similar to the gargoyles on the 61st floor; they were bright and showed no sign of pitting. In the 1954 inspection, some light rust spots had been found on the coping which were apparently caused by the splashing of some foreign material. As was assumed in the former report, these spots had disappeared with normal weathering and the pitting was almost indiscernible.

Empire State Building, New York, N. Y.

This building was erected in 1931 and made use of large amounts of type 302 stainless steel on the exterior. The pilasters on the tower were examined from the 89th floor level; these had never been cleaned and there was no trace of corrosion on the south and west sides and only faint traces of pits on the east and north sides. The surfaces, in general, seemed to have retained their original mill finish. On the 85th floor observation level, the railing which was type 302 and added in 1949 showed no evidence of corrosion. Where the railing was easily accessible to visitors, it was bright and shiny. In less accessible places there was a light film of dirt on the surface but no pitting or corrosion under the dirt.

The type 302 No. 6 finish mullions were examined on all sides of the building on the sixth-floor level. There was a dark adherent dirt on the surface of the mullions that was more prevalent at sheltered parts of the building. Underneath this dirt there were a few light shallow pits which did not give any evidence of having worsened since the last inspection in 1954.

From all indications, there has been practically no deterioration of the stainless steel since its erection 29 years ago. From a distance, in spite of adhering dirt, the stainless steel shines brightly, particularly on sunny days.

Socony Mobil Building, New York, N. Y.

This building was erected in 1955 and with the exception of the glass area of the windows is completely surfaced with type 302 stainless steel panels, 0.038 in. thick. These panels contain a press-formed trihedral pattern to prevent waviness and to break up reflections. Considerable stainless steel is also used in the lobby and elevators.

The stainless steels around the elevators and on the control panels are lacquered to reduce finger spotting. These are cleaned nightly and show no evidence of any corrosion or pitting. The stainless steel at other locations in the lobby is not lacquered. It is cleaned nightly and appears to be in perfect condition.

The first four floors of the exterior, up to an offset, are cleaned twice a year by a cleaning contractor. Columns on the 42nd floor are cleaned by the building employees once a year. The rest of the building has not been cleaned since 1956.

The exterior was examined at the fourth-floor level. From a distance of about 15 ft, no evidence of discoloration could be noticed. Closer inspection revealed small amounts of discoloration on some panels. This discoloration was more noticeable at certain locations and was thought to be associated with rainfall and wind currents. The most discoloration was encountered on the east side near the south corner, but even here it was quite light. It will probably not become worse with time. The window frames on all sides were in excellent condition and showed no signs of rusting, pitting, or discoloration. A few panels showed evidence of incomplete removal of the cleaning compound used to clean the building after erection, but these areas did not seem to be adversely affected. The railing around the 4th floor level is made of type 302 stainless steel and is cleaned about every three months with pumice and whiting. It was in perfect condition.

The columns on the 42nd floor level were in perfect condition except for three columns which contained some light tarnishing on their eastern sides.

As a whole this building is in excellent condition and from a distance sparkles brightly in the sunshine.

Inland Steel Building, Chicago, Ill.

This nineteen story building was built in 1958. There are no interior columns in the main building. The outer surface is composed of about 70 per cent glass and 30 per cent type 202 stainless steel. A service tower, completely enclosed with type 202 stainless steel, houses the elevators and other service equipment.

The stainless steel on the main building exterior is cleaned about once monthly by automatic window washing equipment. The stainless in this area is perfect. The connection between the main building and the tower and most of the tower are cleaned yearly with a proprietary cleaner. The yearly cleaning had been done about one month prior to the inspection. These areas were in very good condition and close inspection failed to show any indications of pitting or corrosion. One side of the tower faced an alley and had never been cleaned. There was a very heavy accumulation of dirt and oily smudge. This area, particularly near the ground level, is used extensively for testing new types of cleaners. There were no pits or corrosion products under this dirt.

Other Stainless Steel Uses in Chicago, Ill.

Stainless steel used for display windows and entrances for several stores were examined. Some were magnetic and presumed to be type 430 stainless steel and others were non-magnetic and thought to be type 302. The appearance of the stainless steel was directly related to cleaning. Where cleaning was frequent the stainless steel was in excellent condition. Where cleaning was very infrequent, slight pits and superficial rusting could be found. This rusting and pitting was thought to be caused by salt used to melt the snow on the streets. In no case was the pitting or rusting so bad that cleaning would not restore the original attractive surface to the stainless steel.

525 William Penn Place Building, Pittsburgh, Pa.

The spandrel panels and windows of this building, which was erected in 1950–1951, are fabricated of type 302 stainless steel. The patterned spandrels were stamped from No. 2B finish sheet and the windows were formed from No. 2B finish sheet or No. 2 finish strip. The interior of the building contains large amounts of No. 4 finish sheet, some type 430 and some type 302. The exterior was last cleaned in 1958.

The type 302 cap flashing on the roof, 41st floor level, was in excellent condition. There was no sign of rust or pits and there was very little dirt on the metal. On the 17th floor level the building was inspected out of open windows. The west and south sides were almost free of dirt accumulation on the spandrel members. On the north and east sides some small amount of dirt had accumulated on the spandrels. There was no indication of rust spots or pitting on the spandrels. Small amounts of dirt had accumulated on the window sills under the bottom sash.

At the 9th floor level the spandrel members showed no rust spots but traces of light pits were found under the light dirt which had collected on the spandrels. Large amounts of dirt had collected on the window sills, under which there were traces of rusting and a few small pits. A sample of the dirt at one of these windows was taken and later analysis revealed that it contained 0.81 per cent chlorides.

The elevators, outside entrances, and the lower eight feet of the paneled lobby are cleaned weekly. The rest of the lobby is cleaned yearly with a proprietary cleaner. The metal showed no signs of deterioration in this location and was in perfect condition.

Gateway Buildings, Pittsburgh, Pa.

Buildings Nos. 2 and 3 were completed in 1952 and building No. 1 was completed in 1953. The entire exterior surfaces are of type 430, No. 2 dull finish cold rolled sheet or strip steel. About two months prior to this inspection, these buildings had been cleaned for the first time since just after erection. Inspection of the sides was possible only from the roof and from the ledge at the second floor. From close inspection at both of these locations on building No. 2 not one sign of rust or pitting could be detected. There was practically no accumulation of dirt on the surface. The metal on these buildings had the appearance of having been "just made."

The entrances and doorways were also clean and showed no traces of rust or discoloration. The store fronts and street level facia were also in very good condition.

Main Boiler House, Irvin Works of U.S. Steel Co., Dravosburg, Pa.

This building, erected in 1938, is approximately 150 by 70 ft, and 60 ft high to the eaves. The roof and sides, both inside and outside, are covered with corrugated stainless steel sheets varying in gage from 18 to 29. Between the inside and outside sheets is a $\frac{3}{8}$ in. thick insulation. There are a few type 430 sheets in the building but most of the sheets are austenitic stainless steel of various compositions and finishes. The building has never been cleaned and is entirely covered with dirt and grime to the extent that there is little metallic luster to the sides.

Examination of the surface after removing the dirt from several spots on the outside of the building revealed some very shallow pits in sheltered areas but no attack on the boldly exposed metal. At one location inside the building there were some rust spots, which were believed to have been caused by some salt which had been stored there, but had since been moved elsewhere. In the rest of the interior the stainless steel sheets showed no signs of corrosion.

The corrugated sides were fastened to the steel structure with cadmium-plated steel screws, lead washers adjacent to the sheet, and galvanized steel washers between the lead washers and the head of the screw. Most of these have corroded to the extent that they have been replaced and the replacements are stainless steel. It was interesting to note that the stainless steel panels for about 1 in. around the remaining galvanized bolts and washers had collected very little dirt.

National City Bank Building, Chester and E. 40th St., Cleveland, Ohio

This building was completed in 1954 and makes use of a considerable amount of type 302, No. 2B finish, around the windows and entrances. The vaults were made of a magnetic stainless thought to be type 410.

The stainless steel on the inside of the building is kept clean and is in excellent condition. The outside of the building has not been cleaned since the superficial cleaning shortly after erection. Some dirt has accumulated around the windows and there are several places where dirt streaks have been caused by drain water. Finger marks are noticeable around the doorways. There is no rusting, pitting, or any evidence of corrosion. Cleaning would restore all the natural beauty of the metal.

Manufacturing Plant Building of E. F. Hauserman Co., Cleveland, Ohio

The sides of this plant are made of insulated panels made from draw bench sections. They are bolted to a steel framework so that they may be easily moved when alterations are necessary. Additions have been made several times and the panels have been relocated and additional panels added; some original panels, stored until needed, were used. This plant is located in the Cleveland flats in a heavy industrial atmosphere. The original building was erected in 1950 using type 301 panels. Later, additions were made using some type 430 panels.

Around some of the entrances there were some superficial rust spots and the Hauserman personnel recognize that this is due to the salt used in the winter months around the entrances. Otherwise, this building which has never been cleaned shows no sign of corrosion. The type 430 panels are slightly darker than the type 301 panels but both seem to have an indefinite life in this building.

Mechanical Shop Building of Cuyahoga Works, American Steel and Wire Division, U. S. Steel Co., Cleveland, Ohio

This building was erected in 1954 and the sides are made of corrugated type 430 stainless steel. It is located in a very dirty industrial atmosphere and has never been cleaned. With the exception of very heavy dirt deposits, the general appearance is quite satisfactory. Close inspection did reveal some superficial rust with very fine pits underneath. The gable end of the building exhibited a slight trace of a peculiar stained condition. We were informed that this had been much more noticeable shortly after erection and was thought to have been caused by iron pick-up from the corrugating rolls.

Miami Beach Federal Savings and Loan Assn., Miami Beach, Fla.

This building, erected in 1956, incorporates type 316 No. 4 finish stainless steel trim around enameled steel panels. The window frames are also made of No. 4 finish type 316 stainless steel. The building is located about 1300 ft west of the beach. The carbon steel screws used to hold the panels rusted badly and were all replaced with type 316 stainless steel screws when the building was last cleaned in March, 1958.

Some very light staining was noticeable on the east and north sides of the building when inspected from a distance of less than five feet, particularly around window screws in sheltered areas. The south side of the building, which is subject to more washing by the rain showed practically no staining except on the underside of the top of the window frames where there were a few spots around screws. From the sidewalk, the building appeared in perfect condition.

The bank entrance, which is cleaned weekly, appeared to be in perfect condition and showed no sign of staining or pitting.

Other Stainless Applications in Miami Beach, Fla.

Flamingo Theatre.—Located about 1200 ft from the ocean. The entrances and sign boards, erected in 1946, are made of type 302 stainless steel, No. 4 finish. They are obviously cleaned regularly and are in perfect condition.

Carib Theatre.—Erected about 1950 and located about 1000 ft from the ocean. The entrance, billboards and marquee are trimmed with No. 4 finish type 302 stainless steel. They are cleaned regularly and are in perfect condition. Mullions on the face of the building above the marquee show considerable brown stain and the management stated that they never clean them but that the staining does not seem to be worsening.

Lee's Health Bar.—Located about 600 ft from the ocean. A sign over the marquee is constructed of stainless steel. It shows considerable staining and seems to be in the same condition as described in a private company report, dated 1955. The present manager reported that he had been operating the business for the past three years and the sign had not been cleaned in that time and he doubted if it had ever been cleaned.

Neon Reflector Signs at Hotels Along Collins Road.—This street is located about 600 ft from the ocean and many hotels use stainless steel for reflectors of neon signs; staining was very apparent. The management of one hotel, Sagamore Hotel, 1675 Collins Road, stated that the sign was 11 years old and had never been cleaned because, in spite of the staining, the reflector continued to do a satisfactory job.

Indian River Power House of Orlando Power Commission, Indian River, Fla.

This building is constructed of type 302, No. 2B finish, corrugated stainless steel panels. All of the stainless was erected in 1959. The plant is located about 800 ft from the brackish Indian River, beyond which is the more brackish Banana River, and then the ocean. The panels had been cleaned a few at a time starting in February, 1959, and finishing in the spring of 1960. Panels known to have been cleaned in February, 1959 had the same appearance as panels cleaned in 1960. At distances of over 25 ft, no discoloration could be seen but upon close inspection, there was considerable staining typical of chloride atmospheres. This condition will probably not become any worse. Considerable oily dirt had accumulated on parts of the building and probably came from the chimney. This dirt will probably be a greater cause for detracting from the appearance of the building than the chloride stains, but even this will probably not be noticeable from the public highway, about 300 ft away.

CONCLUSIONS

1. Stainless steel, in architectural applications, retains its metallic luster and appearance beneath urban dirt and grime. With even limited cleaning there is no deterioration of the metal.

2. In industrial buildings, where appearance is not a big factor, stainless steel has practically an indefinite life, even without cleaning. Some shallow pits and light rust stains may be expected when chlorides are present but the appearance of the building will be governed mainly by adhering dirt.

3. Even after long periods of neglect or abuse, stainless steel can be returned to a pleasing appearance by cleaning. Some slight pitting may then be discernible upon close inspection, the amount depending on the chlorides in the atmosphere and the type of stainless steel.

4. The use of cleaners containing chlorides should be discouraged. Incomplete removal of such cleaners will cause pitting and unsightly rusting and complete removal from crevices is most difficult.

5. Applications of stainless steel in structures not subject to periodic cleaning should, as near as possible, be designed so as not to include crevices which may collect dirt. This dirt may hold some chlorides which can lead to superficial pitting.

6. In environments containing chlorides, stainless steels seem to develop a limited number of rust spots on boldly exposed areas beyond which no further deterioration in appearance proceeds. The amount of rusting depends on the environment and the type of stainless steel. Type 316 stainless steel is the preferred grade for seacoast applications. The apparent cessation of rusting is thought to be associated with the cleaning action of the elements, the maximum being reached when the elements remove the chlorides at the same rate at which they are deposited.

7. The stainless steels are entirely adequate for architectural applications.

The selection of the type of stainless should be governed by the environment, the desired maintenance of appearance, and the planned cleaning program.

The following were members of the task group: R. Doughton, Jr., A. C. Hamstead, C. P. Larrabee, J. H. McConnell, J. S. Pettibone, W. G. Renshaw, and G. M. Reigel.

Respectfully submitted on behalf of the task group,

C. R. MAYNE,
Chairman.

REPORT OF COMMITTEE B-1

ON

WIRES FOR ELECTRICAL CONDUCTORS*

Committee B-1 on Wires for Electrical Conductors held three meetings during the year: in Washington, D. C., on October 25, 1960; in Philadelphia, Pa., on January 24, 1961; and in Richmond, Va., on March 28, 1961. Meetings of Subcommittees II, IV, VI, and VII also were held during the year.

The committee notes with regret the retirement during the year of the following long-time members who have contributed much to the committee's work: D. R. Brobst, C. H. Jensen, W. F. Markley, H. H. Rodee, E. G. Sturdevant, and L. H. Winkler. During the year, C. E. Ambelang resigned as chairman of Subcommittee VI due to impending retirement, and R. E. Koontz was appointed to this office.

The committee consists of 84 members, of whom 44 are classified as producers, 26 as consumers, and 14 as general interest members.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Method of Test for Electrical Conductivity by Use of Eddy Currents (B 342 – 60 T):[1]

Appendix.—Delete the second and third sentences in the present Appendix, and replace them with the following three sentences:

> The eddy-current method measures the average resistance to the flow of eddy currents, within the volume of metal in which there is sensible induction of eddy currents, in planes parallel to and near to the surface. The thickness of this volume, which for some instruments ranges from 0.040 to 0.120 in., varies with the resistivity. The paths of the eddy currents are circular and have components only in planes parallel to the test surface, and not in a direction perpendicular to this surface.

Tentative Definitions of Terms Relating to Uninsulated Metallic Electrical Conductors (B 354 – 60 T):[2]

New Definitions.—Add the following ten new definitions in alphabetical order:

Annealed Wire.—See *Soft Wire.*

Bare Conductor.—A conductor having no nonmetallic covering.

Circular Mil.—A unit of area equal to $\pi/4$ (0.7854....) of a square mil. The cross-sectional area of a circle in circular mils is therefore equal to the square of its diameter in mils. A circular inch is equal to one million circular mils.

Conductor Core.—The center strand or member about which one or more layers of wires or members are laid helically to form a concentric-lay or rope-lay conductor.

Member.—A group of wires stranded together for combination with other stranded groups into a multiple-membered conductor.

Soft Wire.—Wire that has been drawn or rolled to final size and then heated to remove the effects of cold working.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] 1960 Supplement to Book of ASTM Standards, Part 3.

[2] 1960 Supplement to Book of ASTM Standards, Part 2.

Stranded Conductor.—A conductor composed of a group of wires, usually twisted, or of any combination of such groups of wires.

Tinned Wire.—See *Coated Wire.*

Volume Resistivity.—The resistance in ohms of a body of unit length and unit cross-sectional area.

Weight Resistivity.—The resistance in ohms of a body of unit weight and unit length.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Method of Test for Resistivity of Electrical Conductor Materials (B 193 – 60):[1]

Table II.—Delete the group of data now headed aluminum alloy GS10B and replace with the following:

Material	Density, δ, at 20 C, g per cu cm	Temperature Coefficient of Resistance, α, at 20 C
Aluminum alloy 6101, per cent IACS:		
59.5	2.70	0.00393
59.0	2.70	0.00390
57.0	2.70	0.00377
56.0	2.70	0.00370
55.0	2.70	0.00363

Standard Specifications for Rolled Aluminum Rods (EC Grade) for Electrical Purposes (B 233 – 60):[3]

Section 4(c).—Change the present "12,000 psi" to read "11,000 psi."

Standard Specifications for Standard Nominal Diameters and Cross-Sectional Areas of Awg Sizes of Solid Round Wires Used as Electrical Conductors (B 258 – 57):[3]

Section 4.—Change to read as follows:

4. Standard nominal diameters of Awg sizes of solid round wires shall be calculated in accordance with the conventional mathematical law of the American Wire Gage and in accordance with Section 3.

For wire sizes 4/0 to 44 Awg, inclusive, nominal diameters shall be expressed in no more than four significant figures but in no case closer than the nearest 0.1 mil (0.0001 in.).

For wire sizes 45 to 56 Awg, inclusive, nominal diameters shall be expressed to the nearest 0.01 mil (0.00001 in.).

The standard nominal diameters expressed in mils have been calculated in accordance with these rules and are given in Table I for convenient reference (Note 2).

Table I.—Delete the present data for sizes 45 to 50 Awg, inclusive, and replace with data for sizes 45 to 56 Awg, inclusive, as follows:

Size, Awg	Diameter, mils	Cross-Sectional Area	
		circular mils	sq in.
45	1.76	3.10	0.00000243
46	1.57	2.46	0.00000194
47	1.40	1.96	0.00000154
48	1.24	1.54	0.00000121
49	1.11	1.23	0.000000968
50	0.99	0.980	0.000000770
51	0.88	0.774	0.000000608
52	0.78	0.608	0.000000478
53	0.70	0.490	0.000000385
54	0.62	0.384	0.000000302
55	0.55	0.302	0.000000238
56	0.49	0.240	0.000000189

Explanatory Note.—Change Note 2 to read as follows:

NOTE 2.—Awg numbers appearing in columns 1 and 5 of Table I are given only to facilitate conversion from Awg numbers to the wire size in mils. It is emphasized that this is not intended to be an endorsement of the use of Awg numbers to designate wire sizes. Wire diameters should be specified in mils as shown in Table I, columns 2 and 6.

Micrometer calipers calibrated to measure 0.1 mil (0.0001 in.) should be considered satisfactory for measuring the diameters of 4/0 to 44 Awg (0.4600 to 0.0020 in.) inclusive.

For greater accuracy in obtaining the mean diameter of ultrafine wire sizes 45 to 56 Awg (0.00176 to 0.00049 in.) inclusive, the Method of Test for Diameter by Weighing of Fine Wire Used in Electronic Devices and Lamps (ASTM

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[3] 1958 Book of ASTM Standards, Part 2.

Designation: F 205) should be considered satisfactory.

Standard Specifications for Three-Quarter Hard Aluminum Wire for Electrical Purposes (B 262 – 56):[3]

Section 5(b).—Change to read as follows:

(*b*) When requested by the purchaser, tension tests of specimens containing joints made in the finished wire or during the final drawing, as permitted in Section 10(*b*), shall be made. Such tests shall show the tensile strength to be not less than 11,000 psi for electrical butt-welded joints, and not less than 15,000 psi for cold pressure-welded joints.

Standard Specification for Half-Hard Aluminum Wire for Electrical Purposes (B 323 – 60):[3]

Section 5(b).—Change to read as follows:

(*b*) When requested by the purchaser, tension tests of specimens containing joints made in the finished wire or during the final drawing, as permitted in Section 10(*b*), shall be made. Such tests shall show the tensile strength to be not less than 11,000 psi for electrical butt-welded joints, and not less than 13,000 psi for cold pressure-welded joints.

STANDARDS CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following standards:

Standard Specifications for:

Medium-Hard-Drawn Copper Wire (**B 2 – 52**),
Bronze Trolley Wire (**B 9 – 55**),
Copper Trolley Wire (**B 47 – 55**),
Hot-Rolled Copper Rods for Electrical Purposes (**B 49 – 52**),
Hard-Drawn Copper Alloy Wires for Electrical Conductors (**B 105 – 55**),
Figure-9 Deep-Section Grooved and Figure-8 Copper Trolley Wire for Industrial Haulage (**B 116 – 55**),
Hard-Drawn Copper Covered Steel Wire (**B 227 – 57**),
Concentric-Lay-Stranded Copper Covered Steel Conductors (**B 228 – 56**), and
Concentric-Lay-Stranded Copper and Copper Covered Steel Composite Conductors (**B 229 – 56**).

EDITORIAL CHANGES

The committee recommends editorial change as follows in the Standard Specifications for Hard-Drawn Aluminum Wire for Electrical Purposes (**B 230 – 60**):[3]

Explanatory Note.—Change the second paragraph of Note 2 to read as follows:

The maximum volume resistivity of aluminum conductor has been prescribed in Section 7 and this is equivalent to a minimum conductivity of 61.0 per cent of that of the International Annealed Copper Standard (IACS). The corresponding maximum weight resistivity values for aluminum are 0.076397 ohm-gram/meter^2 and 436.24 ohm-lb/mile^2.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Editorial and Records (A. A. Jones, chairman) continued its editorial review of specifications and coordinated the various subcommittee recommendations regarding revisions of specifications.

Subcommittee II on Methods of Test and Sampling Procedures (R. H. Lloyd, chairman) completed revisions of Specifications B 193 and B 342 as noted earlier in the report. A task group is reconsidering suitable methods of test for determining softness of copper wire.

Subcommittee IV on Conductors of Copper and Copper Alloys (M. V. Yokelson, chairman) completed revision of Specification B 258 as noted earlier in the report.

Task groups are cooperating with the U. S. Army Signal Corps Engineering Laboratories in completing a long-time study of gray-tin transformation; reviewing possible need for revision or expansion of tensile properties requirements for alloy wires in Specification B 105; reviewing the possibility of establishing solderability requirements for tin solder-coated copper wires; and reviewing the need for permitting unit joints in members of rope-lay copper conductors.

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee VI on Composite Conductors (R. E. Koontz, chairman) has task groups working on the preparation of specifications for aluminum-clad steel both solid wire and strand and of specifications for copper-covered steel wires for electronic applications.

Subcommittee VII on Conductors of Light Metals (A. L. Kolb, chairman) completed the revisions of Specifications B 233, B 262, and B 323 as noted earlier in the report, and also completed a clarifying editorial revision of Specification B 230.

Task groups are reconsidering the inspection requirements in the various aluminum rod and wire specifications; considering proposed additions and deletions of constructions in Specification B 232; investigating the effects of various in-process welds on finished steel core wire properties; reconsidering recommended package sizes particularly for composite steel-aluminum conductors in cooperation with the Aluminum Assn.; and preparing drafts of new specifications covering certain classes of aluminum-alloy conductors and also compacted strand conductors.

Joint Task Groups.—The joint task group considering the preparation of standard test report forms for bare electrical conductors was discharged because of evident lack of interest. The joint task group on Glossary of Terms is considering additional definitions for future inclusion in Definitions B 354. It is proposed to establish a new joint task group on Industry-Military Specifications to attempt to coordinate military and industry requirements for essentially the same product, with particular regard to inspection and acceptance.

This report has been submitted to letter ballot of the committee, which consists of 84 members; 57 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

D. HALLORAN,
Chairman.

A. A. JONES,
Secretary.

REPORT OF COMMITTEE B-2
ON
NON-FERROUS METALS AND ALLOYS*

Committee B-2 on Non-Ferrous Metals and Alloys met on June 29, 1960, in Atlantic City, N. J. Meetings of the Advisory Committee were held on June 29, 1960 in Atlantic City and on Jan. 6, 1961, in New York City.

The committee consists of 137 members, of whom 63 are classified as producers, 50 as consumers, and 24 as general interest members.

A new subcommittee, designated Subcommittee X on Zirconium and Hafnium, was formed. W. W. Stephens was appointed chairman and F. R. Lorenz, secretary.

A. Y. Bethune replaced Sidney Rolle as chairman of Subcommittee II. George Hiers replaced G. H. Clamer as chairman of Subcommittee III, and Robert Redelfs replaced D. W. Pettigrew as chairman of Subcommittee IV. R. R. Freeman was appointed secretary of Subcommittee VIII.

The committee deeply regrets to report the deaths of Sidney Rolle on March 25 and Ernest E. Thum on April 10. Mr. Rolle served the ASTM and Committee B-2 for many years with devotion and distinction. He was nominated this year by Committee B-2 for the ASTM Annual Award of Merit. Mr. Thum likewise was an outstanding member, having served as secretary of the Committee from 1928 to 1939 and chairman from 1939 to 1948. He has been since that time honorary vice-chairman.

The secretariat of the ISO Committee 26 on Copper was relinquished by the United States to Germany. The working group on raw copper remains under U. S. auspices. A Tentative Specification on Slab Zinc prepared by ISO TC/18 on zinc has been received. On the latter committee the U. S. has observer status.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1960 Annual Meeting, Committee B-2 presented to the Society through the Administrative Committee on Standards the recommendations listed below. The recommendations were accepted by the administrative committee on the dates indicated.

New Tentative Specifications:

Zirconium and Zirconium Alloy Forgings and Extrusions for Nuclear Applications (B 356 – 60 T) (Accepted Sept. 29, 1960),
Seamless and Welded Unalloyed Titanium Welding Fittings (B 363 – 61 T) (Accepted Feb. 15, 1961),
Tantalum Ingots and Flat Mill Products (B 364 – 61 T) (Accepted March 1, 1961),
Tantalum Rod and Wire (B 365 – 61 T) (Accepted March 1, 1961),
Factory-Made Wrought Nickel and Nickel-Base Alloy Welding Fittings (B 366 – 61 T) (Accepted May 10, 1961), and
Titanium and Titanium Alloy Castings (B 367 – 61 T) (Accepted May 10, 1961).

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

Revisions of Tentative Specifications:

Solder Metal (B 32 - 60 T) (Accepted Dec. 20, 1960),
Seamless Nickel and Nickel Alloy Condenser and Heat Exchanger Tubes (B 163 - 58 T) (Accepted July 12, 1961),
Nickel-Chromium-Iron Alloy Rod and Bar (B 166 - 58 T) (Accepted July 12, 1961),
Nickel-Chromium-Iron Alloy Seamless Pipe and Tube (B 167 - 58 T) (Accepted July 12, 1961), and
Nickel-Chromium-Iron Alloy Plate, Sheet, and Strip (B 168 - 58 T) (Accepted July 12, 1961).

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for:

White Metal Bearing Alloys (Known Commercially as "Babbitt Metal") (**B 23 - 59 T**),[1]
Nickel-Copper Alloy Plate, Sheet, and Strip (**B 127 - 58 T**),[2]
Nickel Rod and Bar (**B 160 - 58 T**),[2]
Nickel Seamless Pipe and Tube (**B 161 - 58 T**),[2]
Nickel Plate, Sheet, and Strip (**B 162 - 58 T**),[2]
Nickel-Copper Alloy Rod and Bar (**B 164 - 58 T**),[2] and
Nickel-Copper Alloy Seamless Pipe and Tube (**B 165 - 58 T**).[2]

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for more than six years without revision:

Standard Specifications for:

Lake Copper Wire Bars, Cakes, Billets, Ingots, and Ingot Bars (**B 4 - 42**),
Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (**B 5 - 43**),
Nickel (**B 39 - 22**),
Electrolytic Cathode Copper (**B 115 - 43**),
Fire-Refined Copper for Wrought Products and Alloys (**B 216 - 49**), and
Metallic Antimony (**B 237 - 52**).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Specifications for:

Titanium and Titanium Alloy Strip, Sheet, and Plate (**B 265 - 58 T**),
Titanium Sponge (**B 299 - 58 T**),
Secondary Lead (**B 325 - 58 T**),
Seamless and Welded Titanium Pipe (**B 337 - 58 T**), and
Titanium and Titanium Alloy Bars and Billets (**B 348 - 59 T**).

Tentative Classification of:

Pig Tin (**B 339 - 59 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Refined Copper (H. Barkell, chairman).—A proposed tentative specification on deoxidized billets is under review to resolve minor objections. A joint task group with Committees B-1 on Electrical Conductors and B-5 on Copper and Copper Alloys is reviewing the Classification of Coppers (B 224 - 58).

Subcommittee II on Lead (A. Y. Bethune, chairman). — Specification B 29 - 55 for Pig Lead is being reviewed by a task group to resolve an objection to the wording of the scope clause. Another task group has the responsibility of preparing a tentative specification for lead for nuclear applications.

Subcommittee III on Tin-Base and Lead-Base Alloys (G. O. Hiers, chairman) has decided to defer the proposed Symposium on Solders until 1962. A task group has been appointed to study a specification for paste-type solder.

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[1] Available as a separate reprint.
[2] 1958 Book of ASTM Standards, Part 2.
[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee IV on Refined Zinc and Wrought Zinc (R. Redelfs, chairman).—A task group is continuing work on a specification for cast and wrought zinc anodes for cathodic protection.

Subcommittee VIII on Miscellaneous Metals and Alloys (R. I. Jaffee, chairman).—A task group formulating molybdenum specifications should soon submit four tentative specifications covering mill products. Another task group is preparing four tentative specification covering columbium mill products. The beryllium task group is preparing tentative specifications for four forms of beryllium. The tantalum task group is considering the possible inclusion of electron-beam grade tantalum in its specifications. The tungsten task group is preparing three specifications for tungsten products. A request has been received for the subcommittee to investigate the need for specifications covering the rare metals. There is nothing to report from task groups on sodium and thorium.

Subcommittee IX on Titanium and Titanium Alloys (A. F Jones, chairman) expects to offer a revision of the Tentative Specification for Seamless and Welded Unalloyed Titanium Tubing (B 338 - 58 T). A tentative specification for titanium and titanium alloy forgings is being prepared.

Subcommittee X on Zirconium and Hafnium (W. W. Stephens, chairman) has been organized recently. Tentative Specification for Wrought Zirconium and Zirconium Alloy Seamless and Welded Tubes for Nuclear Service (B 353 - 60 T) is now being revised. A task group will continue preparation of a tentative specification on zirconium and zirconium alloy forgings and extrusions for nuclear applications.

This report has been submitted to letter ballot of the committee, which consists of 137 members: 96 members returned their ballots, of whom 90 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

ALFRED BORNEMANN,
Chairman.

C. K. CONARD,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee B-2 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specification for:

Titanium Alloy Forgings (B 381 - 61 T).

Revision of Tentative Specification for:

Seamless and Welded Unalloyed Titanium Tubing (B 338 - 58 T).

These recommendations were accepted by the Standards Committee on August 9, 1961, and the new and revised tentative specifications appear in the 1961 Book of ASTM Standards, Part 2.

REPORT OF COMMITTEE B-3
ON
CORROSION OF NON-FERROUS METALS AND ALLOYS*

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys held two meetings during the past year: in Atlantic City, N. J., on June 27, 1960, and in Cincinnati, Ohio, on Jan. 30, 1961.

The committee consists of 89 members, of whom 82 are voting members; 30 are classified as producers; 27 as consumers, and 25 as general interest members.

NEW TENTATIVE

The committee recommends for publication as tentative the Method of Copper-Accelerated Acetic Acid - Salt Spray (Fog) Testing (CASS Test), as appended hereto.[1]

ADOPTION OF TENTATIVES AS STANDARD WITH EDITORIAL CHANGES

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard with editorial changes as indicated:

Tentative Method of Salt Spray (Fog) Testing (B 117 - 57 T):[2]

Section 6.—In the last sentence, correct the spelling of "electrometrically" and insert the words "at 25 C (77 F)" after the word "electrometrically."

Change Note 1 of this section to read: "A solution having a specific gravity of 1.0255 to 1.0400 at 25 C (77 F) will meet the concentration requirement. It is suggested that a daily check be made."

Section 8(b).—In Note 2, change the first sentence to read: "A solution having a specific gravity of 1.0255 to 1.0400 at 25 C (77 F) will meet the concentration requirement." Change the last sentence to read: "A solution that requires between 3.4 and 5.1 ml of 0.1 *N* silver nitrate will meet the concentration requirements."

Tentative Method of Acetic Acid - Salt Spray (Fog) Testing (B 287 - 57 T):[2]

Section 6.—In the next to last sentence, add the words "25 C (77 F)" after the word "electrometrically."

Change Note 1 of this section to read: "A solution having a specific gravity of 1.0255 to 1.0400 at 25 C (77 F) will meet the concentration requirement. It is suggested that a daily check be made."

Section 8(b).—In Note 2, change the first sentence to read: "A solution having a specific gravity of 1.0255 to 1.0400 at 25 C (77 F) will meet the concentration requirement." Change the last sentence to read: "A solution that requires between 3.4 and 5.1 ml of 0.1 *N* silver nitrate will meet the concentration requirements."

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] The new tentative appears in the 1961 Book of ASTM Standards, Parts 3 and 8.

[2] 1958 Book of ASTM Standards, Parts 3 and 8.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Method of:

Total Immersion Corrosion Test of Non-Ferrous Metals (**B 185 – 43 T**), and
Alternate Immersion Corrosion Test of Non-Ferrous Metals (**B 192 – 44 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee III on Salt Spray (C. O. Durbin, chairman).—Since the Salt Spray (Fog) Test (B 117 – 57 T) and the Acetic Acid - Salt Spray (Fog) Test (B 287 – 57 T) have been used successfully without major change for several years, the subcommittee recommends that both methods be adopted as standard. The subcommittee recommends the publication of a new Tentative Method of Copper-Accelerated Acetic Acid - Salt Spray (Fog) Testing (CASS Test) appended to this report. This method is primarily applicable to the rapid testing of decorative copper-nickel-chromium or nickel-chromium coatings on steel and zinc-base die castings designed for relatively severe service. The test is also used on buffed or electropolished decorative stainless steel for similar service.

Work is also in progress on a method of corrosion testing of decorative chromium plating by the clay-pack high humidity (Corrodkote) procedure. The Corrodkote test is conducted by application of a slurry containing corrosive salts to test specimens, allowing the slurry to dry, and exposing the test specimens coated with the slurry to a high relative humidity for a specified period of time. The Corrodkote test is primarily applicable to copper-nickel-chromium or nickel-chromium plated parts.

Subcommittee VI on Atmospheric Corrosion (J. S. Pettibone, chairman).—Testing of the 1-yr exposure specimens (aluminum only) has been completed. A task group is collating these data for publication as an appendix to the Committee B-3 Annual Report in the 1961 *Proceedings*.[4]

The 2-yr exposure specimens have been removed from test, color-photographed, and returned to the original contributors for testing.

Subcommittee VII on Weather (C. P. Larrabee, chairman).—A program for measuring various atmospheric exposure factors such as specimen wetness time, amounts of oxides of sulfur, and changes of temperature is under way at sites at Trail, B. C.; Ottawa, Ont.; Cleveland, Ohio; South Bend, Pa.; and Kure Beach, N. C. These data will be compared with weight loss of steel, zinc, and copper.

A 3-yr program is under way at 39 U. S. and foreign sites for the determination of their atmospheric corrosivities in terms of steel and zinc.

Subcommittee VIII on Galvanic and Electrolytic Corrosion (G. V. Kingsley, chairman).—A task group expects to have a report covering 1-yr exposure data on the magnesium panel-type galvanic couple tests ready for the Annual Meeting.[5]

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[4] See p. 205.

[5] See p. 215.

This report has been submitted to letter ballot of the committee which consists of 82 voting members; 62 members returned their ballots of whom 57 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

K. G. COMPTON,
Chairman.

W. H. AILOR,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee B-3 presented to the Society through the Administrative Committee on Standards the recommendation that the Method of Corrosion Testing of Decorative Chromium Plating by the Corrodkote Procedure be published as tentative. This recommendation was accepted by the Standards Committee on August 9, 1961, and the new tentative method appears in the 1961 Book of ASTM Standards, Part 3, bearing the designation B 380 – 61 T.

APPENDIX I

REPORT OF SUBCOMMITTEE VI ON ATMOSPHERIC CORROSION

1957 TEST PROGRAM—ONE-YEAR EXPOSURES

In its original concept, the 1957 test program of Subcommittee VI called for scheduled removal periods at 2, 7, and 20 yr (triplicate panels for exposure and duplicate panels for control). This program was modified in consideration of the characteristic corrosion-rate curve for aluminum to include a 1-yr removal period for the 34 aluminum test alloys. The details of the entire test program were published in Appendix I to the Committee B-3 Annual Report for 1959.[1]

The present report is concerned with the data obtained from the examination of specimens removed after 1-yr exposure and control panels of these 34 aluminum alloys plus three additional alloys tested under the same program requirements by the Aluminium Development Assn. at five test sites in England. One alloy (super-purity aluminum, alloy code No. 87) supplied by the Aluminium Development Assn. is under test at the four ASTM sites and the five sites in England.

ALUMINUM ALLOY SPECIMENS

The 1957 program contains 34 aluminum alloys on test at four ASTM sites and three additional aluminum alloys on test at five English locations. The number of aluminum alloys grouped by major alloying element is as follows:

Aluminum (99.00 per cent minimum and greater)	4
Copper	8
Manganese	3
Silicon	1
Magnesium	13
Magnesium and silicon	4
Zinc	4

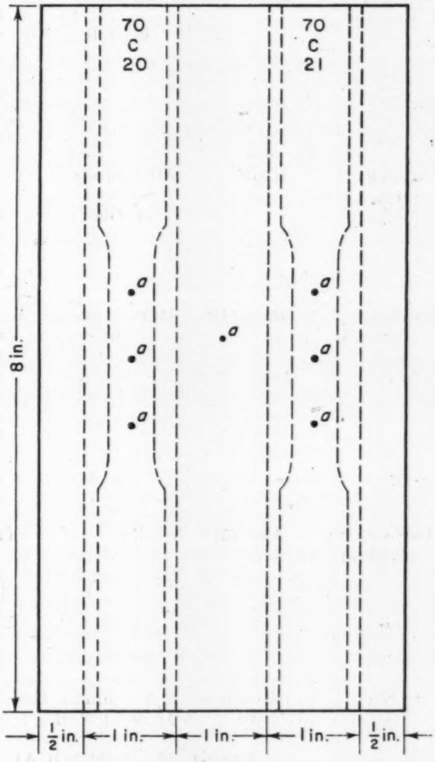

FIG. 1.—Exposure Panel, Indicating Location of Tension Test Bars and Marking Code.

The code indicates: Alloy number at top, test site location letter, and specimen number. Points designated *a* are the points at which thickness measurements are made.

The aluminum alloys were supplied, tested and evaluated after exposure by the following companies:

Aluminum Company of America

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 59, p. 176 (1959).

TABLE I.—THE BRITISH TEST SITES.

Location	Site Code	Classification	Description
Sheffield, England	*S* (1)[a]	Severe Industrial	Located approximately 1 mile NE of center of city in heavy industrial area on roof of Firth Brown Laboratories (60 + ft above ground). Ringed by industrial chimneys at radius of approximately 200 yd and has one small chimney and two chemical laboratory fume chimneys on the roof itself. Specimens face SW[b] (direction of prevailing winds).
London, England	*L* (2)[a]	Moderately Severe Industrial	In a residential - light industrial section at Euston, London, on roof of British Non-Ferrous Metals Research Association laboratory building (40 + ft above ground). Atmospheric pollution from nearby buildings, the B.N.F.M.R.A. adjacent chemical laboratory and foundry, and the nearby Euston railroad station. Specimens face SW[b] (direction of prevailing winds).
Hayling Island, England	*H* (3)[a]	Moderately Severe Marine	In a boatyard at the head of an inlet on the eastern side of Hayling Island. Located so that specimens are exposed about 4 ft above normal high tide level and the water runs under them for about 4 hr each tide. Remote from any forms of atmospheric pollution. Specimens face south[b] (toward the water).
Banbury, England	*Ban.* (4)[a]	Rural (some pollution)	Located 1 to 2 miles north of Banbury in rural surroundings. On a grassy plot (10 by 60 yd) to southwest of Aluminium Laboratories Ltd. Research Center. Experimental foundry and other laboratories adjacent to northwest and railway line 75 yd to southward. Plant buildings of Northern Aluminium Co. are ½ mile to east. Atmospheric SO_2 at site indicate pollution about 1/10 that observed in London. Specimens face SW[b] (direction of prevailing winds).
Anglesey, England	*Ang.* (5)[a]	Marine	Ground level, on property of Saunders Roe Ltd., Beaumaris, Anglesey, about 30 yd from high water mark. Water lies to southeast and forms part of straits between Anglesey and mainland. Site is free from industrial pollution and it is reported that salt spray mist is present in winter in the neighborhood of test specimens. Specimens face SW[b].

[a] Number in parentheses is Aluminium Development Association site code.

[b] All specimens are at an angle of 30 deg from the horizontal.

TABLE II.—CHEMICAL ANALYSES OF ALUMINUM ALLOYS EXPOSED IN ENGLAND.

Alloy Code Commercial designation	No. 1[a] Super-Purity	No. 2 NS6-¼ Hard	No. 3 HS30-WP	No. 4 HC15-WP
Copper, per cent	0.0015	<0.04	0.21	4.88
Iron, per cent	0.0035	0.27	0.40	0.37
Silicon, per cent	0.0025	0.18	0.96	0.80
Manganese, per cent	0.0005	0.35	0.52	0.77
Magnesium, per cent	...	4.95	0.69	0.65
Zinc, per cent	...	0.06	...	0.02
Chromium, per cent	...	...	0.02	<0.02
Titanium, per cent	...	...	...	...
Nickel, per cent	...	...	...	...
Aluminum	Remainder	Remainder	Remainder	Remainder

[a] ASTM alloy code No. 87.

Aluminium Development Assn. (laboratory testing by the British Non-Ferrous Metals Research Assn.)
Kaiser Aluminum and Chemical Corp.
Reynolds Metals Co.

Test Periods—Data from the 1-yr exposure (triplicate panels) and 1-yr storage (duplicate panels) are summarized in Table IV. Future removals are scheduled for the 2-, 7-, and 20-yr periods.

Test Panels, 8 by 4 by 0.05 in. were marked in accordance with Fig. 1 by alloy code number (arbitrarily assigned to each alloy in the program), site location code letter, and panel specimen number. The four alloys exposed in England (including alloy code No. 87), were identified by a system of holes punched near the panel edges. The panel areas were corrected for the changes caused by the holes.

TABLE III.—FIRST EXPOSURE PERIOD FOR THE ALUMINUM ALLOYS.

Location	Site Code	Date Exposed	Date Removed	Exposure Days
Kure Beach (80-ft. site), N. C.	A	Nov. 22, 1958	Nov. 21, 1959	364
Newark, N. J. (New York Area)	B	Oct. 31, 1958	Nov. 20, 1959	385
Point Reyes, Calif.				
Alloys 10 to 36	C	May 24, 1958	July 22, 1959	424
Alloys 37 to 39 and 85 and 86		June 11, 1958	July 22, 1959	406
Alloy 87		Jan. 22, 1959	July 22, 1959	192
State College, Pa.	D	Nov. 6, 1958	Nov. 5, 1959	364
Sheffield, England	S (1)[a]	Aug. 26, 1959	Aug. 10, 1960	350
London, England	L (2)[a]	Aug. 7, 1959	Aug. 8, 1960	367
Hayling Island, England	H (3)[a]	Aug. 28, 1959	Aug. 24, 1960	362
Banbury, England	Ban. (4)[a]	Aug. 11, 1959	Aug. 11, 1960	366
Anglesey, England	Ang. (5)[a]	Sept. 29, 1959	Aug. 13, 1960	380

[a] Number in parentheses is site code of Aluminium Development Assn. (British).

TEST PROGRAM

Test Site Locations:

Site *A*—Kure Beach (80-ft site), N. C. (east coast marine atmosphere); Site *B*—New York Area (Newark, N. J.) (industrial); Site *C*—Point Reyes, Calif. (west coast marine); and Site *D*—State College (University Park), Pa. (rural). The British test locations are: Site *S*—Sheffield (industrial); Site *L*—London (industrial); Site *H*—Hayling Island (marine); Site *Ban.*—Banbury (rural); and Site *Ang.*—Anglesey (marine). Since descriptions of the ASTM test sites are given in the literature,[2] only the British test locations are shown in Table I.

Laboratory Tests Prior to Exposure:

Chemical Analyses were made on all 34 test alloys as reported in Appendix I to the 1959 Report of Committee B-3.[1] The analyses of the three other alloys are shown in Table II, along with that of the super-purity alloy, code No. 87 (as reported in the above report).

Thickness Measurements were made to the nearest 0.001 in. on the specific locations shown in Fig. 1.

Length and Width Measurements were made to the nearest 0.01 in.

Weighing of the aluminum alloys was to the nearest 0.001 g.

Tension Tests were made on six specimens, three each from two panels.

Evaluation of Corrosion Damage:

Cleaning of all exposed and storage aluminum panels was accomplished by use of a 10-min immersion in a solution

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[2] Report of the Advisory Committee on Corrosion, *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 229 (1958).

TABLE IV.—ONE-YEAR DATA, 1957 TEST PROGRAM, BY ALLOY AND EXPOSURE SITE FOR ALUMINUM ALLOYS.

Alloy Code	Commercial Designation	Density	Panel Numbers	Days Exposed	Site[a]	Weight Change, mg per dm sq	Corrosion Rate		Pit Depth, mil (4 Deepest)			Tensile Strength, psi		Elongation in 2 in., per cent	
							mg per dm sq per day	mg per yr	Sky	Ground	Avg	Control	Exposed	Control	Exposed
No. 10	1188-H14	2.70	73 to 78	364	A	5.4436	0.0150	0.0079	1.5	0.9	1.2	11 950	11 650	12.5	11.4
			79 to 84	385	B	9.4616	0.0246	0.0130	1.6	1.0	1.2		11 850		12.5
			85 to 90	424	C	3.0400	0.0072	0.0038	1.2	0.5	0.9		11 700		12.0
			91 to 96	364	D	2.2660	0.0062	0.0033	0.3	0.5	0.4		11 833		12.6
No. 11	1135-H14	2.70	73 to 78	364	A	11.2822	0.0310	0.0164	1.3	1.6	1.5	16 430	16 517	11.8	11.4
			79 to 84	385	B	10.2725	0.0267	0.0141	1.7	1.2	1.5		16 550		12.3
			85 to 90	424	C	7.9277	0.0187	0.0099	1.3	0.9	1.1		16 633		10.8
			91 to 96	364	D	2.7942	0.0077	0.0041	0.4	0.3	0.4		16 300		12.8
No. 12	3004-H34	2.70	73 to 78	364	A	9.9723	0.0274	0.0144	2.2	1.8	2.0	36 420	36 250	8.8	8.7
			79 to 84	385	B	23.2856	0.0631	0.0333	1.3	2.9	2.1		36 267		8.3
			85 to 90	424	C	5.0222	0.0119	0.0063	1.0	[b]	0.5		36 083		8.8
			91 to 96	364	D	3.8545	0.0106	0.0056	[b]	0.1	0.1		36 550		9.0
No. 13	4043-H14	2.70	73 to 78	364	A	12.5872	0.0346	0.0179	2.0	0.9	1.0	22 600	22 367	11.4	11.3
			79 to 84	385	B	11.7183	0.0305	0.0161	1.6	0.7	1.1		22 117		11.3
			85 to 90	424	C	10.6727	0.0252	0.0133	1.1	1.0	1.1		22 450		10.1
			91 to 96	364	D	2.9838	0.0073	0.0038	0.3	0.2	0.2		22 767		10.0
No. 14	X5454-0	2.68	73 to 78	364	A	10.2885	0.0283	0.0151	1.5	2.5	2.0	36 920	37 183	20.4	20.9
			79 to 84	385	B	18.5365	0.0482	0.0257	1.4	2.6	2.0		36 900		21.5
			85 to 90	424	C	6.6287	0.0156	0.0083	1.3	0.5	0.9		37 200		21.7
			91 to 96	364	D	5.2366	0.0144	0.0077	[b]	0.2	0.1		37 067		21.6
No. 15	X5454-H34	2.76	73 to 78	364	A	8.9264	0.0245	0.0131	1.6	2.2	1.9	45 200	45 900	8.9	8.6
			79 to 84	385	B	16.9465	0.0444	0.0237	1.7	2.0	1.9		45 733		8.8
			85 to 90	424	C	5.6860	0.0134	0.0070	0.7	[b]	0.4		45 983		8.6
			91 to 96	364	D	5.1421	0.0141	0.0075	0.1	0.1	0.1		45 817		8.8
No. 16	Alclad 5155-H34	2.66	73 to 78	364	A	9.2027	0.0253	0.0137	1.5	0.9	1.1	51 430	52 567	10.2	10.3
			79 to 84	385	B	11.3651	0.0295	0.0159	1.5	1.7	1.5		52 083		10.1
			85 to 90	424	C	4.7448	0.0112	0.0060	0.6	0.2	0.3		52 033		10.3
			91 to 96	364	D	3.0200	0.0083	0.0045	0.1	[b]	0.1		52 350		10.0

No. 17	5456-O	2.64	73 to 78	364	A	10.7585	0.0296	0.0161	1.4	0.8	1.1	49 380	50 117	21.9	21.6
			79 to 84	385	B	18.4254	0.0479	0.0261	2.1	2.8	2.4		50 200		21.7
			85 to 90	424	C	6.2406	0.0147	0.0104	2.1	0.9	1.1		49 880		22.1
			91 to 96	364	D	6.2145	0.0171	0.0100	0.3	0.1	0.2		50 167		21.8
No. 18	1100-H14	2.71	73 to 78	365	A	12.63	0.0347	0.0185	1.6	1.4	1.5	[c]	18 417	[c]	7.4
			79 to 84	386	B	14.55	0.0377	0.0198	1.7	1.6	1.7		18 233		7.8
			85 to 90	425	C	7.62	0.0180	0.0097	1.1	0.9	1.0		18 417		7.3
			91 to 96	365	D	2.83	0.0078	0.0042	[b]	[b]	[b]		18 400		7.6
No. 19	2014-T4	2.80	73 to 78	365	A	36.85	0.1008	0.0520	3.7	1.3[d]	2.8	[c]	68 323	[c]	17.4
			79 to 84	386	B	46.75	0.1210	0.0622	1.8[d]	1.7[d]	1.8		66 933		17.7
			85 to 90	425	C	54.25	0.1275	0.0655	1.5[d]	3.5	2.5		66 233		13.3[e]
			91 to 96	365	D	5.40	0.0150	0.0075	[b]	[b]	[b]		69 300		20.6
No. 20	Alclad 2014-T6	2.80	73 to 78	365	A	11.47	0.0315	0.0160	0.0	0.7	0.6	[c]	70 233	[c]	9.4
			79 to 84	386	B	13.07	0.0338	0.0173	0.9	0.6	0.8		69 583		9.2
			85 to 90	425	C	6.98	0.0165	0.0085	0.6	0.4	0.5		69 623		9.4
			91 to 96	365	D	2.88	0.0085	0.0040	[b]	[b]	[b]		69 567		9.4
No. 21	Alclad 2024-T3	2.77	73 to 78	365	A	8.70	0.0240	0.0125	0.4	0.6	0.5	[c]	62 033	[c]	17.8
			79 to 84	386	B	10.70	0.0277	0.0145	1.0	0.4	0.7		66 200		18.8
			85 to 90	425	C	7.30	0.0173	0.0090	0.2	1.4	0.8		64 683		18.4
			91 to 96	365	D	2.20	0.0060	0.0030	[b]	[b]	[b]		65 817		18.0
No. 22	3003-H14	2.73	73 to 78	365	A	10.88	0.0297	0.0158	1.4	0.6	1.0	[c]	21 817	[c]	6.9
			79 to 84	386	B	19.92	0.0515	0.0270	1.1	1.1	1.1		21 800		7.4
			85 to 90	425	C	9.35	0.0220	0.0117	1.1	[b]	0.6		21 950		6.9
			91 to 96	365	D	2.68	0.0073	0.0038	[b]	[b]	[b]		21 950		7.0
No. 23	5005-H34	2.69	73 to 78	365	A	10.83	0.0297	0.0158	1.1	0.9	1.0	[c]	33 450	[c]	6.4
			79 to 84	386	B	14.17	0.0367	0.0197	1.3	1.6	1.5		33 423		6.9
			85 to 90	425	C	6.22	0.0145	0.0077	1.2	0.2	0.7		22 500		6.5
			91 to 96	365	D	2.45	0.0067	0.0035	[b]	[b]	[b]		22 183		6.9
No. 24	5050-H34	2.69	73 to 78	365	A	10.43	0.0285	0.0152	0.8	1.0	0.9	[c]	27 750	[c]	6.3
			79 to 84	386	B	11.73	0.0305	0.0163	1.3	1.4	1.4		27 700		6.5
			85 to 90	425	C	5.68	0.0133	0.0070	1.1	2.3	1.8		27 817		6.3
			91 to 96	365	D	2.65	0.0072	0.0038	[b]	[b]	[b]		27 817		6.3
No. 25	5052-H34	2.68	73 to 78	365	A	8.50	0.0232	0.0125	0.5	0.7	0.6	[c]	35 667	[c]	10.0
			79 to 84	386	B	13.35	0.0345	0.0187	1.2	1.1	1.2		35 500		9.4
			85 to 90	425	C	4.33	0.0068	0.0055	1.2	[b]	0.6		35 600		9.6
			91 to 96	365	D	2.05	0.0057	0.0032	[b]	[b]	[b]		35 717		9.9

TABLE IV.—ONE-YEAR DATA, 1957 TEST PROGRAM, BY ALLOY AND EXPOSURE SITE FOR ALUMINUM ALLOYS—*Continued*

Alloy Code	Commercial Designation	Density	Panel Numbers	Days Exposed	Site[a]	Weight Change, mg per dm sq	Corrosion Rate		Pit Depth, mil (4 Deepest)			Tensile Strength, psi		Elongation in 2 in., per cent	
							mg per dm sq per day	mg per yr	Sky	Ground	Avg	Control	Exposed	Control	Exposed
No. 26	5083-O	2.65	73 to 78	365	A	14.33	0.0392	0.0217	1.3	0.3	0.8	[c]	44 833	[c]	21.0
			79 to 84	386	B	19.42	0.0503	0.0273	1.5	2.6	2.1		44 800		20.3
			85 to 90	425	C	6.25	0.0147	0.0080	0.6	[b]	0.3		45 017		21.4
			91 to 96	365	D	5.13	0.0142	0.0078	[b]	[b]	[b]		45 433		21.3
No. 27	5086-H34	2.66	73 to 78	365	A	9.27	0.0253	0.0138	[b]	[b]	[b]	[c]	48 533	[c]	10.0
			79 to 84	386	B	19.73	0.0512	0.0277	1.0	1.4	1.2		48 100		10.5
			85 to 90	425	C	5.17	0.0122	0.0065	0.3	1.3	0.8		48 233		10.4
			91 to 96	365	D	3.83	0.0105	0.0057	[b]	[b]	[b]		48 500		10.5
No. 28	7075-T6	2.80	73 to 78	365	A	35.98	0.0987	0.0508	1.8[d]	1.0	1.4	[c]	81 867	[c]	9.6
			79 to 84	386	B	22.23	0.0577	0.0295	0.9[d]	2.0[d]	1.5		81 800		8.4
			85 to 90	425	C	33.00	0.0775	0.0398	1.3	1.6[d]	1.5		81 583		8.6
			91 to 96	365	D	4.27	0.0118	0.0060	[b]	[b]	[b]		82 017		9.8
No. 29	Alclad 7075-T6L	2.80	73 to 78	365	A	10.98	0.0302	0.0122	1.2	1.1	1.2	[c]	78 417	[c]	11.4
			79 to 84	386	B	12.22	0.0317	0.0162	1.4	0.9	1.3		76 383		10.7
			85 to 90	425	C	9.17	0.0217	0.0112	0.4	1.2	0.8		76 233		10.6
			91 to 96	365	D	1.00	0.0027	0.0014	[b]	0.7	0.4		75 800		10.7
No. 30	2024-T3	2.77	73 to 78	364	A	37.4148	0.1028	0.0533	2.3	2.7	2.5	69 700	69 900	18.8	18.6
			79 to 84	385	B	40.0054	0.1039	0.0598	0.9	0.4	0.7		69 500		18.1
			85 to 90	424	C	36.4762	0.0860	0.0446	2.7	5.7	4.2		68 100[e]		13.6
			91 to 96	364	D	12.5721	0.0346	0.0179	0.1	[b]	0.1		70 200		19.2
No. 31	2024-T36	2.77	73 to 78	364	A	31.7882	0.0873	0.0452	1.7	2.5	2.1	74 600	70 500	13.8	8.6
			79 to 84	385	B	41.5925	0.1080	0.0560	1.0	0.8	0.9		71 200		11.9
			85 to 90	424	C	44.8180	0.1057	0.0548	2.0	5.8	3.9		69 800		7.2
			91 to 96	364	D	15.5044	0.0426	0.0220	0.1	0.3	0.2		74 400		13.2
No. 32	2024-T81	2.77	73 to 78	364	A	39.1593	0.1076	0.0558	2.1	1.7	1.9	69 800	69 900	6.0	4.0
			79 to 84	385	B	55.0819	0.1431	0.0742	1.1	0.7	0.9		69 300		5.4
			85 to 90	424	C	48.0750	0.1134	0.0587	2.1	4.1	2.1		68 800		4.1
			91 to 96	364	D	11.9894	0.0329	0.0171	0.1	1.0	0.6		70 800		5.8

No. 33.......	2024-T86	2.77	73 to 78	364	A	43.4631	0.1194	0.0619	1.8	1.8	1.8	75 200	75 700	5.0	3.3
			79 to 84	385	B	50.9084	0.1322	0.0685	0.8	0.9	0.9		76 500		4.7
			85 to 90	424	C	59.2007	0.1396	0.0724	2.0	3.7	2.9		75 400		2.8
			91 to 96	364	D	17.5421	0.0482	0.0250	0.1	0.4	0.3		77 400		5.1
No. 34.......	Alclad 3003-H14	2.73	73 to 78	364	A	10.6280	0.0292	0.0153	4.7	3.9	4.3	22 100	21 700	5.6	6.2
			79 to 84	385	B	12.2883	0.0319	0.0168	1.1	0.5	0.8		21 700		6.4
			85 to 90	424	C	8.5190	0.0201	0.0105	3.2	4.5	3.9		21 800		5.6
			91 to 96	364	D	2.9786	0.0065	0.0034	b	b	b		21 500		6.8
No. 35.......	5154-H34	2.66	73 to 78	364	A	7.9999	0.0220	0.0118	1.7	1.4	1.6	43 700	43 200	10.2	10.1
			79 to 84	385	B	15.7980	0.0410	0.0221	0.7	0.6	0.7		43 400		10.3
			85 to 90	424	C	4.1729	0.0099	0.0053	0.2	2.9	1.6		43 400		10.3
			91 to 96	364	D	3.3114	0.0091	0.0049	b	b	b		43 200		9.9
No. 36.......	5357-H34	2.70	73 to 78	364	A	6.5848	0.0181	0.0096	0.6	0.6	0.6	24 700	24 700	7.5	7.5
			79 to 84	385	B	12.0393	0.0313	0.0133	b	b	b		24 600		7.5
			85 to 90	424	C	3.1026	0.0073	0.0038	0.7	1.2	1.0		24 800		7.8
			91 to 96	364	D	2.3837	0.0066	0.0035	b	b	b		24 800		7.5
No. 37.......	6061-T4	2.70	73 to 78	364	A	12.4582	0.0342	0.0181	0.2	b	0.1	39 900	39 900	19.1	18.8
			79 to 84	385	B	18.1403	0.0471	0.0250	1.0	0.7	0.9		40 400		19.5
			85 to 90	406	C	8.2451	0.0203	0.0108	0.6	2.3	1.5		40 200		18.9
			91 to 96	364	D	3.8516	0.0106	0.0056	0.3	b	0.2		41 500		20.0
No. 38.......	6061-T6	2.70	73 to 78	364	A	14.9640	0.0411	0.0218	b	0.9	0.5	46 700	46 800	10.8	10.4
			79 to 84	385	B	19.5116	0.0507	0.0269	0.9	0.5	0.7		46 900		10.3
			85 to 90	406	C	12.6566	0.0312	0.0165	1.0	2.8	1.9		46 800		10.8
			91 to 96	364	D	3.3648	0.0092	0.0049	b	b	b		47 300		11.1
No. 39.......	Alclad 6061-T6	2.70	73 to 78	364	A	11.5610	0.0317	0.0168	0.5	b	0.3	44 700	44 400	11.7	11.5
			79 to 84	385	B	12.8656	0.0334	0.0178	1.1	0.7	0.9		44 500		11.5
			85 to 90	406	C	13.2345	0.0326	0.0173	1.3	1.8	1.6		44 900		11.7
			91 to 96	364	D	2.2819	0.0063	0.0033	b	b	b		45 000		12.0
No. 84.......	5083-H34	2.65	73 to 78	365	A	7.47	0.0203	0.0110	b	b	b	c	54 000	c	11.8
			79 to 84	386	B	23.00	0.0597	0.0325	0.6	0.4	0.5		52 833		11.2
			85 to 90	406	C	5.60	0.0138	0.0075	0.5	b	0.3		54 067		11.9
			91 to 96	365	D	2.12	0.0058	0.0033	b	b	b		54 300		11.6
No. 85.......	7079-T6	2.80	73 to 78	364	A	12.5301	0.0344	0.0177	1.5	1.5	1.5	77 950	77 600	11.7	11.4
			79 to 84	385	B	20.8506	0.0542	0.0278	1.3	1.5	1.4		77 117		11.6
			85 to 90	406	C	12.8131	0.0316	0.0155	2.0	1.6	1.8		77 000		11.4
			91 to 96	364	D	5.1415	0.0142	0.0073	b	b	b		77 617		11.5

TABLE IV.—ONE-YEAR DATA, 1957 TEST PROGRAM, BY ALLOY AND EXPOSURE SITE FOR ALUMINUM ALLOYS—*Continued*

Alloy Code	Commercial Designation	Density	Panel Numbers	Days Exposed	Site[a]	Weight Change, mg per dm sq	Corrosion Rate		Pit Depth, mil (4 Deepest)			Tensile Strength, psi		Elongation in 2 in., per cent	
							mg per dm sq per day	mg per yr	Sky	Ground	Avg	Control	Exposed	Control	Exposed
No. 86	Alclad 7079-T6	2.80	73 to 78	364	A	10.8706	0.0298	0.0153	0.6	0.9	0.8	72 350	72 600	11.2	10.8
			79 to 84	385	B	12.1813	0.0316	0.0166	1.0	0.6	0.8		72 550		11.2
			85 to 90	406	C	9.9487	0.0245	0.0126	0.5	0.5	0.5		72 217		10.8
			91 to 96	364	D	3.0068	0.0082	0.0043	0.2	0.1	0.1		72 317		11.0
No. 87	1199-H18	2.70	73 to 78	364	A	4.6362	0.0128	0.0067	2.6	3.4	3.0	16 000	16 100	5.9	6.1
			79 to 84	385	B	10.1140	0.0263	0.0139	0.9	1.2	1.1		15 900		6.2
			85 to 90	192	C	1.7651	0.0099	0.0052	2.0	2.1	2.1		16 100		5.5
			91 to 96	364	D	2.2011	0.0060	0.0032	[b]	[b]	[b]		16 200		5.2
No. 87	SI-H	2.70	11 to 13	350	S	197.9	0.566	0.301	2.7	4.9	3.8	16 100	15 420	4.8[f]	3.7
			23 to 25	367	L	81.8	0.223	0.119	2.6	4.5	3.6		15 480		3.8
			35 to 37	362	H	10.3	0.029	0.015	1.7	2.0	1.8		15 730		5.3
			47 to 49	366	Ban	6.1	0.017	0.009	0.8	0.4	0.6		15 890		5.3
			121 to 123	380	Ang	6.3	0.016	0.009	1.4	1.6	1.5		15 880		5.7
....	NS6-¼H	2.65	11 to 13	350	S	341.4	0.976	0.529	3.5	5.4	4.5	49 710	44 220	11.0	7.2
			23 to 25	367	L	141.6	0.386	0.210	2.0	2.4	2.2		47 010		8.8
			35 to 37	362	H	27.5	0.076	0.041	1.0	1.6	1.3		48 740		9.5
			47 to 49	366	Ban	14.7	0.040	0.022	1.0	1.0	1.0		49 380		10.5
			121 to 123	380	Ang	22.9	0.060	0.032	0.9	1.7	1.3		48 090		11.5
...	HS30-WP	2.70	11 to 13	350	S	279.4	0.798	0.425	4.2	4.4	4.3	45 420	42 250	10.5	6.7
			23 to 25	367	L	132.4	0.361	0.192	1.7	2.6	2.1		43 540		8.7
			35 to 37	362	H	13.2	0.036	0.019	1.2	1.1	1.2		45 500		10.0
			47 to 49	366	Ban	9.1	0.025	0.013	2.7	0.8	1.8		45 310		10.8
			121 to 123	380	Ang	13.4	0.035	0.019	1.0	1.3	1.2		45 320		10.2

...	HC15-WP	2.79	11 to 13	350	S	318.6	0.910	0.469	2.1	2.3	2.2	65 390	64 870	9.3	9.0
			23 to 25	367	L	142.2	0.388	0.200	2.3	2.5	2.4		64 930		9.0
			35 to 37	362	H	14.9	0.041	0.021	1.6	2.0	1.8		64 810		9.2
			47 to 49	366	Ban	8.1	0.022	0.011	1.5	1.1	1.3		65 590		8.8
			121 to 123	380	Ang	15.0	0.040	0.020	1.7	2.0	1.9		65 080		9.0

[a] A —Kure Beach (80-foot site) N. C.
B —New York Area (Newark, N. J.)
C —Point Reyes, Calif.
D —State College, Pa.
S —Sheffield, England
L —London, England
H —Hayling Island, England
Ban.—Banbury, England
Ang.—Anglesey, England

Symbol Key: [b] No appreciable attack.
[c] Control data not available.
[d] Intergranular attack.
[e] One specimen broke at end of gage marks.
[f] Two specimens broke at end of gage marks.

containing 2 per cent chromic acid and 5 per cent phosphoric acid (by weight) at 180 F, followed by a 2 to 3 min dip in 70 per cent nitric acid. The use of ultrasonics was found to facilitate this procedure.

Weighing of the cleaned panels was to the nearest 0.001 g.

Tension Tests were made on six specimens, three each from duplicate storage panels and two each from triplicate exposure panels after cleaning and weighing.

Pit Depth Determinations were made on the center section of each panel after removal of tension specimens. A dial depth gage was used where feasible with metallographic checks to determine the four deepest pits on both the skyward and groundward sides of the panel. A calibrated eyepiece microscope was used in some cases to make these determinations.

Exposure Periods:

Although the exposure period for this phase of the 1957 test program was approximately 1 yr, there was considerable variation in the actual number of days of exposure as shown in Table III.

The subcommittee is indebted to the following task group for developing and presenting the 1-yr data of the 1957 test program:

W. H. AILOR, *Task Force Chairman*
WILLIAM KING
F. P. PORTER
T. J. SUMMERSON

Respectfully submitted on behalf of the subcommittee,

J. S. PETTIBONE,
Chairman

APPENDIX II

REPORT OF SUBCOMMITTEE VIII ON GALVANIC AND ELECTROLYTIC CORROSION

MAGNESIUM PLATE-TYPE GALVANIC COUPLES—PART III OF A THREE-PART PROGRAM

This study represents the third part of a program to evaluate the galvanic corrosion caused by various dissimilar metals when coupled to magnesium alloys and exposed to different atmospheric conditions. Part I[1] dealt with disk-type couples, Part II[2] with spool and wire couples and Part III with plate-type couples in which the galvanic effect of dissimilar metals could be evaluated through loss in tensile strength of the base panel when compared with similar uncoupled control panels exposed alongside of the coupled panels. The data presented in this report were determined on the first of three sets of panels removed after 1 year's exposure at four different locations.

After much discussion extending over several years and supported by pilot tests, the type of panel illustrated in Fig. 1 was adopted to evaluate by means of loss in tensile strength the galvanic effect of various dissimilar metals when coupled as strips to two magnesium alloys, AZ31B-H24[3] and ZK60A-T5.[3] No protection was applied to any of the components of the couple assembly. Strips *A* and *B* were bolted to both sides of the base panel. A uniform torque of 30 lb-in. was applied to all bolts except the plastic bolts which could be safely tightened to a torque of only 8 lb-in. In some cases $\frac{5}{16}$ in. diameter aluminum alloy rivets were used in place of aluminum alloy bolts. The purpose of strip *A* was to insure a uniform contact pressure between the base panel and strip *B*. Wherever possible, the composition of strip *A* was the same as that of the fastener to eliminate the possible effect of another dissimilar metal in the assembly. This type of panel permitted the machining of tension specimens which could be used to obtain a quantitative evaluation of the galvanic effect of the metals coupled to the magnesium alloys.

Table I lists the combinations of metals that were coupled to both AZ31B-H24 and ZK60A-T5. The analyses of the metals used in this program are given in Table II. The surface finish and thickness of the dissimilar metals are also included in this table. The magnesium-alloy components were degreased and then acid pickled at room temperature to remove 0.0005 in. of each surface in a solution having the following composition:

Glacial acetic acid	25½ fluid oz
Sodium nitrate	6⅔ oz
Water	to make 1 gal

The aluminum-alloy components were

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[1] H. O. Teeple, "Atmospheric Galvanic Corrosion of Magnesium Coupled to Other Metals," Symposium on Atmospheric Corrosion of Non-Ferrous Metals, *ASTM STP No. 175*, Am. Soc. Testing Mats., p. 89 (1955).

[2] Report of Subcommittee VIII on Galvanic and Electrolytic Corrosion, *Proceedings*, Am. Soc. Testing Mats., Vol. 54, p. 150 (1954).

[3] See Standard Specification for Magnesium-Base Alloy Forgings (B 91 – 58), 1961 Book of ASTM Standards, Part 2, p. 1076.

exposed with a commercial finish after vapor degreasing. The mild steel, stainless steel, monel, and brass components were also vapor degreased and further cleaned, where necessary, to remove any surface contamination in accordance with the standard surface preparation procedures for these metals.

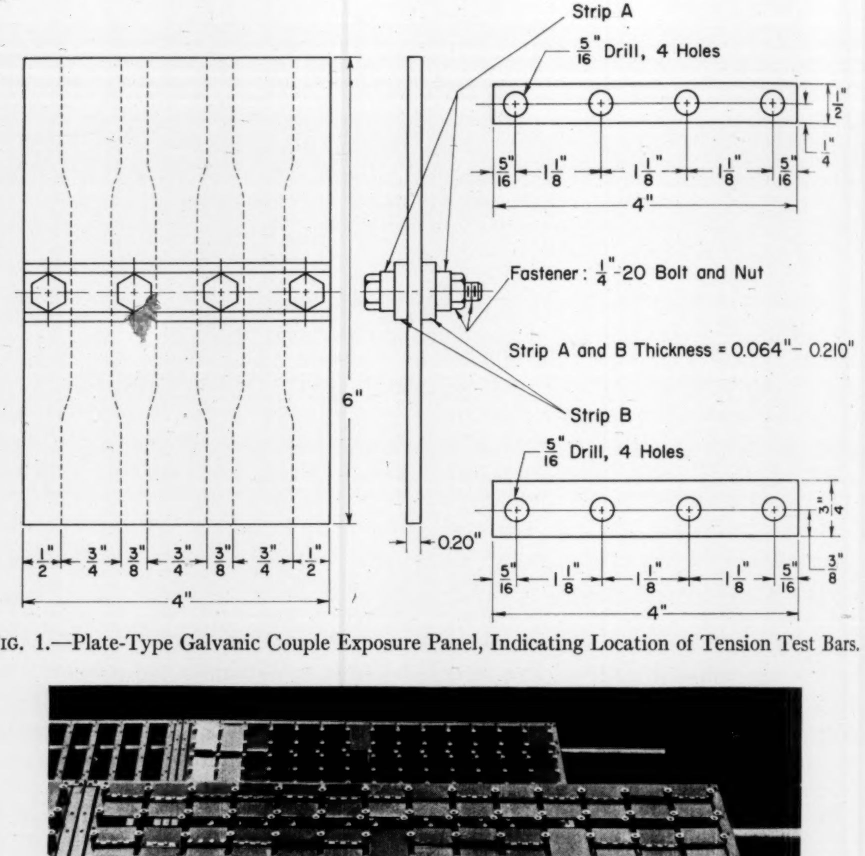
Strip A
5/16" Drill, 4 Holes
1/2"
1/4"
5/16" 1 1/8" 1 1/8" 1 1/8" 5/16"
4"
Fastener: 1/4"-20 Bolt and Nut
Strip A and B Thickness = 0.064"- 0.210"
Strip B
5/16" Drill, 4 Holes
3/4"
3/8"
5/16" 1 1/8" 1 1/8" 1 1/8" 5/16"
4"
6"
0.20"
1/2" 3/4" 3/8" 3/4" 3/8" 3/4" 1/2"
4"

FIG. 1.—Plate-Type Galvanic Couple Exposure Panel, Indicating Location of Tension Test Bars.

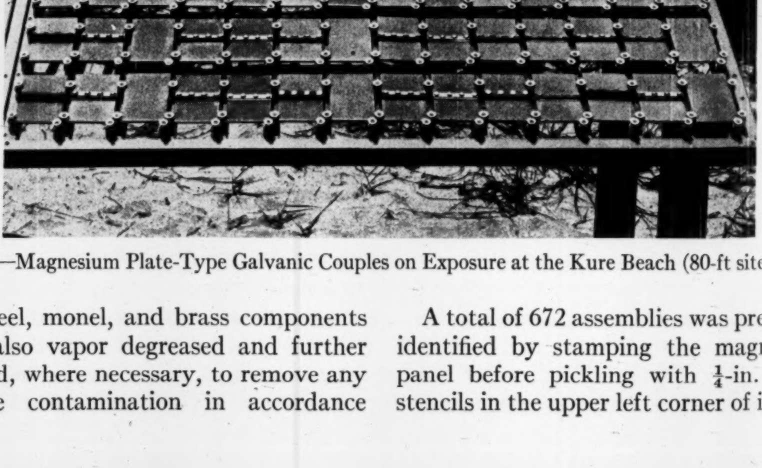

FIG. 2.—Magnesium Plate-Type Galvanic Couples on Exposure at the Kure Beach (80-ft site), N. C.

A total of 672 assemblies was prepared, identified by stamping the magnesium panel before pickling with $\frac{1}{4}$-in. metal stencils in the upper left corner of its skyward surface, as shown in Figs. 3 and 4. The code for identification of the galvanic couple is shown in Table III. After assembly, the couples were distributed as indicated in Tables IV and V. It is expected that the exposure periods at the Kure Beach, N. C., 80-ft site will be 1, 2, and 5 years, and 1, 5, and 10 or more years at the other exposure sites. Table V also reveals the actual exposure period of the first set of 224 panels which were removed after 1 year's exposure at the four locations.

The New York Area (Newark, N. J.) site represents a severe industrial environment; State College, Pa., a clean rural atmosphere; the Kure Beach 80-ft (from the beach) site, a severe marine atmosphere; and the Kure Beach 800-ft site, a milder marine environment. Further description of these locations is given in the 1958 report of the Advisory Committee on Corrosion.[4] The plate-type couples were supported between porcelain insulators on standard atmospheric exposure frames which were inclined at an angle of 30 deg to the horizontal, as shown in Fig. 2.

Shortly after removal of the first set of couples from each location the contact resistance between strip *B* and the base panel was measured to determine electrical continuity. These data are given in Table VI. Except for the panels with the plastic bolts, which crept during exposure at the Kure Beach 80-ft site and thereby caused a loose assembly, good electrical contact was maintained during one year's exposure.

After disassembly of the couples, the magnesium-alloy panels were cleaned for 1 min in a hot (200 F) 20 per cent chromic acid solution to remove all corrosion product. The extent of galvanic attack on each of these panels was then visually evaluated in terms of an arbitrary 1 to 5 scale where 1 represented the least and 5 the greatest amount of galvanic attack. Figures 3 and 4 illustrate the degree of

TABLE I.—METALS COUPLED TO MAGNESIUM ALLOYS AZ31B-H24 AND ZK60A-T5

Specimen	Fastener[a]	Strip A	Strip B
No. 1	6061-T6	6061-T6	99.99 per cent aluminum
No. 2	6061-T6	6061-T6	99.99 per cent aluminum + 2 per cent magnesium
No. 3	6061-T6	6061-T6	5356-H32
No. 4	6061-T6	6061-T6	6061-T6
No. 5	6061-T6	6061-T6	5052-H34
No. 6	6061-T6	6061-T6	Alclad 7075-T6
No. 7	Mild steel	Mild steel	Mild steel
No. 8	Brass	Brass	Brass
No. 9	Type 304 stainless steel	Type 304 stainless steel	Type 304 stainless steel
No. 10	Chrome-plated type 304 stainless steel	Chrome-plated type 304 stainless steel	Chrome-plated type 304 stainless steel
No. 11	Monel	Monel	Monel
No. 12	5056-H32[b]	5356-H32	5356-H32
No. 13	5056-H32[b]	5356-H32	Alcan B54S-O[d]
No. 14	6061-T6	6061-T6	AZ31B or ZK60A[c]
No. 15	Nylon	AZ31B or ZK60A[c]	AZ31B or ZK60A[c]
No. 16	5056-H32[b]	5356-H32	AZ31B or ZK60A[c]
No. 17	None	None	None

[a] $\frac{1}{4}$ in.-20 bolt and nut except as noted.
[b] $\frac{5}{16}$ in. diam button head rivet.
[c] Strip was same composition as base panel.
[d] Similar to 5083 composition.

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[4] Report of Advisory Committee on Corrosion, *Proceedings*, Am. Soc. Testing Mats., Vol 58, p. 229 (1958).

TABLE II.—CHEMICAL ANALYSES OF METALS USED IN MAGNESIUM PLATE-TYPE GALVANIC COUPLE PROGRAM.

Including data on surface finish and thickness

MAGNESIUM ALLOYS

	AZ31B-H24	ZK60A-T5
Aluminum, per cent	2.8	0.01
Calcium, per cent	0.00	0.00
Copper, per cent	0.002	0.002
Iron, per cent	0.000	0.000
Manganese, per cent	0.42	0.055
Nickel, per cent	0.000	0.000
Lead, per cent	0.000	0.006
Silicon, per cent	0.00	0.00
Tin, per cent	0.00	0.00
Zinc, per cent	1.00	5.56
Zirconium, per cent	0.00	0.61
Finish	Pickled	Pickled
Thickness, in.	0.188	0.202

ALUMINUM ALLOYS

	99.99 per cent Aluminum	99.99 per cent Aluminum + 2 per cent Magnesium	5052-H34	B54S-O	5356-H32	6061-T6	Clad 7075-T6	
							Core	Cladding
Copper, per cent	0.001	0.002	0.01	0.025	0.05	0.31	1.69	0.01
Iron, per cent	0.004	0.004	0.12	0.23	0.12	0.12	0.20	0.13
Magnesium, per cent	0.00	1.8	2.41	4.35	4.95	0.94	2.63	0.00
Manganese, per cent	0.00	0.00	0.01	0.30	0.09	0.01	0.00	0.00
Silicon, per cent	0.00	0.00	0.07	0.12	0.08	0.53	0.10	0.07
Zinc, per cent	0.00	0.00	0.00	...	0.00	0.00	5.74	1.00
Chromium, per cent	0.00	0.00	0.25	...	0.12	0.24	0.22	0.00
Titanium, per cent	0.00	0.00	0.00	...	0.13	0.05	0.05	0.00
Finish	[a]	[a]	[a]	[a]	[a]	[a]	[a]	[a]
Thickness, in.	0.064	0.064	0.206	0.190	0.204	0.205	0.205[b]	...

STEEL ALLOYS

	Mild Steel	Type 304 Stainless Steel
Carbon, per cent	0.18	0.07
Copper, per cent	0.05	0.47
Manganese, per cent	0.51	1.6
Phosphorus, per cent	0.010	0.026
Sulfur, per cent	0.032	0.015
Silicon, per cent	0.07	0.64
Chromium, per cent	0.09	18.1
Nickel, per cent	0.03	9.8
Finish	Cold rolled	No. 4[c]
Thickness, in.	0.210	0.064

RED BRASS

Copper, per cent	85.01
Zinc, per cent	14.96 (by difference)
Lead, per cent	0.005
Iron, per cent	0.02
Finish	Cold rolled
Thickness, in.	0.205

TABLE II.—CHEMICAL ANALYSES OF METALS USED IN MAGNESIUM PLATE-TYPE GALVANIC COUPLE PROGRAM—*Continued.*

MONEL (2)[d]	
Nickel, per cent	67.0
Copper, per cent	30.0
Iron, per cent	1.4
Manganese, per cent	1.0
Silicon, per cent	0.1
Carbon, per cent	0.15
Sulfur, per cent	0.01
Finish	Cold rolled
Thickness, in.	0.125

CHROMIUM-PLATED TYPE 304 STAINLESS STEEL[e]	
Solution (120 to 125 F):	
Chromic acid	40 oz per gal
Sulfuric acid	0.4 oz per gal
Current density	200 amp per sq ft
Thickness of plate, in.	approx 0.00002 in.

[a] Commercial finish.

[b] Total thickness of clad material.

[c] No. 4 finish, see Specifications for Corrosion-Resisting Chromium and Chromium-Nickel Steel Plate, Sheet, and Strip for Fusion-Welded Unfired Pressure Vessels (A 240), 1961 Book of ASTM Standards, Part 1.

[d] Nominal analysis.

[e] No analysis made of plated coating. Base metal is same as type 304 stainless steel above.

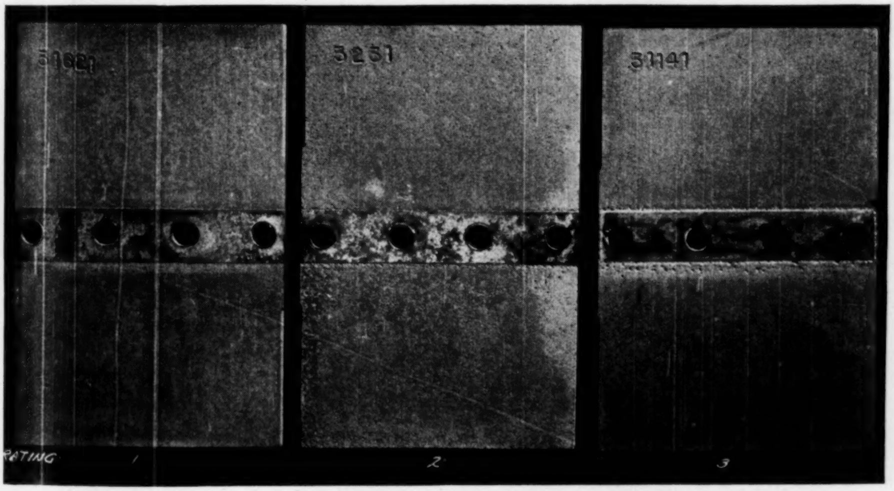

FIG. 3.—Panels Illustrating Galvanic Attack Represented by Ratings 1 to 3.

attack represented by each of the rating numbers.

Three tension test blanks were cut, as indicated in Fig. 1, from the cleaned magnesium galvanic couple panels and were machined into ½ in. wide tension specimens in accordance with Fig. 7 of ASTM Methods E 8,[5] except that the reduced section *A* was shortened to 2 in.

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[5] Methods of Tension Testing of Metallic Materials (E 8 - 61 T), 1961 Book of ASTM Standards, Part 3.

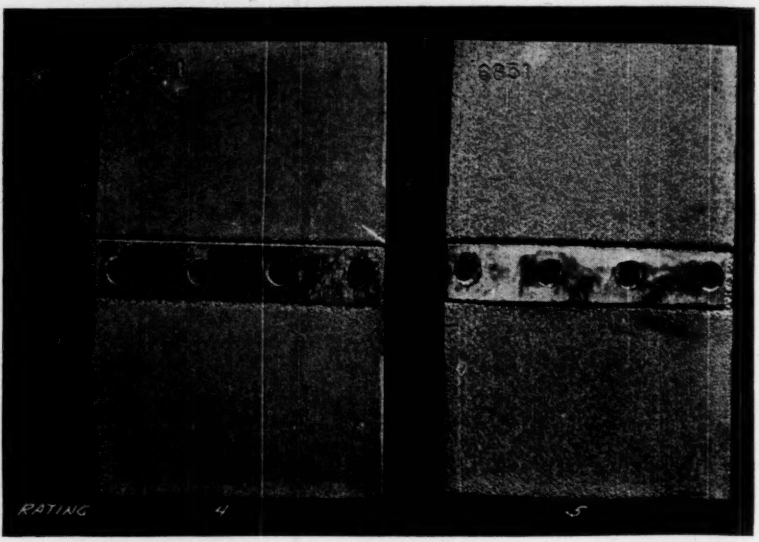

FIG. 4.—Panels Illustrating Galvanic Attack Represented by Ratings 4 and 5.

TABLE III.—CODE FOR IDENTIFICATION OF GALVANIC COUPLES.

First Number: Alloy	3—AZ31B 6—ZK60A
Second Number: Item No.	1 to 17; see Table I
Third Number: Exposure Site	1—New York Area (Newark, N. J.) 2—State College, Pa. 3—Kure Beach, (80-ft site), N. C. 4—Kure Beach (800-ft site), N. C.
Fourth Number: Sample No.	1 to 3 for AZ31B 1 to 6 for ZK60A

Example.—Couple 6831 is one of six ZK60-brass couples exposed at the Kure Beach (80-ft site). N. C.

TABLE IV.—DISTRIBUTION OF EXPOSURE PANELS.

	Number of Combinations	Number of Panels per Exposure Period	Number of Exposure Periods	Number of Exposure Sites	Total Number of Panels
AZ31B couple	16	1	3	4	192
AZ31B control	1	4	3	4	48
ZK60A couple	16	2	3	4	384
ZK60A control	1	4	3	4	48
Grand total					672

TABLE V.—EXPOSURE SITES AND LENGTH OF EXPOSURE OF THE FIRST SET OF MAGNESIUM PLATE-TYPE GALVANIC COUPLES.

Location	Exposure Period	Length of Exposure	
		Days	Years
New York Area (Newark, N. J.)	10-10-58 to 1-25-60	472	1.29
State College, Pa.	9-25-58 to 12-16-59	447	1.22
Kure Beach (80-ft site), N. C.	12-6-58 to 1-12-60	402	1.10
Kure Beach (800-ft site), N. C.	12-6-58 to 1-12-60	402	1.10

TABLE VI.—CONTACT RESISTANCE AFTER 1 YEAR'S EXPOSURE.[a]

Specimen	Dissimilar Metal (Strip B)[b]	AZ31 Panels[c]				ZK60 Panel[d]			
		New York	State College	Kure Beach		New York	State College	Kure Beach	
				80-ft Site	800-ft Site			80-ft Site	800-ft Site
No. 1	99.99 per cent aluminum	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil
No. 2	99.99 per cent aluminum + 2 per cent magnesium	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil
No. 3	5356	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil
No. 4	6061	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil
No. 5	5052	Nil	0.2	Nil	Nil	Nil	Nil	Nil	Nil
No. 6	Alclad 7075	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil
No. 7	Mild Steel	Nil	0.2	0.6	Nil	0.2	Nil	6.3, 0.2	Nil
No. 8	85-15 brass	Nil	0.2	Nil	Nil	0.2	Nil	0.2	Nil
No. 9	Type 304 stainless steel	Nil	Nil	0.2	Nil	Nil	Nil	0.6, 1.8	Nil
No. 10	Chromium-plated type 304 stainless steel	Nil	Nil	Nil	Nil	Nil	Nil	2.8, 33.0	Nil
No. 11	Monel	Nil	Nil	100	Nil	Nil	Nil	0.2, Nil	1.1, 3.5
No. 12	5356	Nil	Nil	Nil	Nil	Nil	Nil	0.8, Nil	Nil
No. 13	Alcan B54S	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil
No. 14	AZ31 (6061 strip A)	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil
No. 15	AZ31 (AZ31 strip A)	Nil	Nil	>100,000[e]	Nil	Nil	Nil	>100,000[e]	Nil
No. 16	AZ31 (5356 strip A)	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil

[a] Resistance measurements are expressed in ohms, and represent the average contact resistance, except where noted, obtained between strip *B* and the base panel on both skyward and groundward surfaces.

[b] Items 1 to 6: Bolts and strip *A* are 6061.
Items 7 to 11: Bolts and strip *A* are same as strip *B* which is in contact with panel.
Items 12, 13, 16: Rivets and strip *A* are 5056 and 5356, respectively.
Item 14: Bolts and strip *A* are 6061.
Item 15: Bolts and strip *A* are plastic and AZ31, respectively.

[c] One panel per test.

[d] Two panels per test.

[e] Creep of plastic bolts resulted in a loose assembly.

because the over-all length of the test specimen was 6 in. The two lines of galvanic attack were at the center of, and at right angles to, the longitudinal axis of the tension specimens. Four tension specimens were machined from each of the magnesium control panels. For all specimens, the original thickness was used for area determinations, and all tension tests were conducted as outlined

TABLE VII.—EFFECT OF VARIOUS DISSIMILAR METALS ON THE GALVANIC CORROSION OF AZ31B.

Galvanic Attack Rating[a]	Exposure Site			
	New York	State College	Kure Beach 80-ft Site	Kure Beach 800-ft Site
1....	AZ31[b] 99.99 per cent aluminum 99.99 per cent aluminum + 2 per cent magnesium 5356[c] 6061 5052 Alclad 7075 Alcan B54S	AZ31[b] 99.99 per cent aluminum 99.99 per cent aluminum + 2 per cent magnesium 5356[c] 6061 5052 Alclad 7075 Alcan B54S Chrome-plated 304 stainless steel	AZ31[b]	AZ31[b] 99.99 per cent aluminum 99.99 per cent aluminum + 2 per cent magnesium 5356[c] 6061 5052 Alcan B54S
2....	Chrome-plated 304 stainless steel	304 stainless steel	99.99 per cent aluminum 99.99 per cent aluminum + 2 per cent magnesium 5356[c] 6061 5052 Alcan B54S	Alclad 7075 Chrome-plated 304 stainless steel
3....	85-15 brass 304 stainless steel Monel	Mild steel 85-15 brass Monel	Alclad 7075 Chrome-plated 304 stainless steel	304 stainless steel Monel
4....	Mild steel		Monel[d] 304 stainless steel[d]	Mild steel 85-15 brass
5....			Mild steel[d] 85-15 brass[d]	

[a] Based upon visual examination after cleaning of panels. (See also Figs. 3 and 4 of cleaned panels showing various degrees of galvanic corrosion.)

1—No significant galvanic corrosion at 5× magnification.
2—Slight galvanic attack; some shallow pits.
3—Moderate attack; scattered medium deep pitting.
4—Moderate attack; continuous band of medium deep pitting.
5—Heavy attack; continuous band of deep pitting.

[b] Includes 3 different combinations of fastener materials:
1. 6061 strip *A* + 6061 bolts.
2. AZ31B strip *A* + plastic bolts.
3. 5356 strip *A* + 5056 rivets.

[c] Includes 2 different combinations of fastener materials:
1. 6061 strip *A* + 6061 bolts.
2. 5356 strip *A* + 5056 rivets.

[d] Galvanic corrosion caused crack near outside bolt hole of base panel.

TABLE VIII.—EFFECT OF VARIOUS DISSIMILAR METALS ON THE GALVANIC CORROSION OF ZK60A.

Galvanic Attack Rating[a]	Exposure Site			
	New York	State College	Kure Beach 80-ft site	Kure Beach 800-ft site
1....	ZK60[b] 99.99 per cent aluminum 99.99 per cent aluminum + 2 per cent magnesium 5356[c] 6061 5052 Alcan B54S Alclad 7075	ZK60[b] 99.99 per cent aluminum 99.99 per cent aluminum + 2 per cent magnesium 5356[c] 6061 5052 Alcan B54S Alclad 7075 Chrome-plated 304 stainless steel	ZK60[b]	ZK60[b]
2....	Chrome-plated 304 stainless steel	304 stainless steel	99.99 per cent aluminum 99.99 per cent aluminum + 2 per cent magnesium 5356[c] 6061 5052 Alcan B54S	99.99 per cent aluminum 99.99 per cent aluminum + 2 per cent magnesium 5356[c] 6061 5052 Alcan B54S Alclad 7075
3....	85-15 brass Monel	85-15 brass Monel Mild steel	Alclad 7075	Chrome-plated 304 stainless steel
4....	304 stainless steel Mild steel		Chrome-plated 304 stainless steel	304 stainless steel 85-15 brass Monel Mild steel
5....			Mild steel[d] 85-15 brass[d] 304 stainless steel[d] Monel[d]	

[a] Based upon visual examination after cleaning of panels. (See also Figs. 3 and 4 of cleaned panels showing various degrees of galvanic corrosion.)

1—No significant galvanic corrosion at 5× magnification.
2—Slight galvanic attack; some shallow pits.
3—Moderate attack; scattered medium deep pitting.
4—Moderate attack; continuous band of medium deep pitting.
5—Heavy attack; continuous band of deep pitting.

[b] Includes 3 different combinations of fastener materials:

1. 6061 strip *A* + 6061 bolts.
2. ZK60A strip *A* + plastic bolts.
3. 5356 strip *A* + 5056 rivets.

[c] Includes 2 different combinations of fastener materials:

1. 6061 strip *A* + 6061 bolts.
2. 5356 strip *A* + 5056 rivets.

[d] Galvanic corrosion caused crack near outside bolt hole of base panel.

TABLE IX.—TENSILE STRENGTH[a] OF AZ31B-H24 PANELS AFTER 1 YEAR'S EXPOSURE AT FOUR SITES.

Specimen	Metal Coupled to AZ31B			New York		State College		Kure Beach 80-ft site		Kure Beach 800-ft site	
	Fastener	Strip A	Strip B	Avg	Range	Avg	Range	Avg	Range	Avg	Range
No. 1...	6061	6061	99.99 per cent aluminum	40 090	210	40 140	870	40 390	360	40 790	110
No. 2...	6061	6061	99.99 per cent aluminum + 2 per cent magnesium	40 520	200	40 100	540	40 040	420	40 510	480
No. 3...	6061	6061	5356	40 070	410	40 430	240	39 950	220	40 660	300
No. 4...	6061	6061	6061	40 170	150	40 600	20	40 100	150	40 590	30
No. 5...	6061	6061	5052	40 020	190	40 710	140	39 850	580	40 310	380
No. 6...	6061	6061	Alclad 7075	39 710	540	40 730	700	38 450	740	40 090	260
No. 7...	Mild steel	Mild steel	Mild steel	37 800	340	39 920	400	35 550	340	38 280	1000
No. 8...	Brass	Brass	Brass	37 660	460	39 780	520	34 400	1790	37 930	960
No. 9...	304 stainless steel	304 stainless steel	304 stainless steel	39 120	840	40 140	230	36 890	210	38 450	610
No. 10...	Plated 304 stainless steel[b]	Plated 304 stainless steel[b]	Plated 304 stainless steel[b]	40 100	170	40 550	160	36 550	140	40 060	290
No. 11...	Monel	Monel	Monel	38 730	600	40 070	160	36 210	1060	38 790	130
No. 12...	5056 rivets	5356	5356	39 750	320	40 410	110	39 850	150	40 800	160
No. 13...	5056 rivets	5356	Alcan B54S	39 430	1520	39 920	390	39 620	300	40 710	200
No. 14...	6061	6061	AZ31B	39 770	250	41 380	130	40 080	510	40 470	180
No. 15...	Plastic	AZ31B	AZ31B	40 350	220	39 580	1160	41 000	310	40 520	210
No. 16...	5056 rivets	5356	AZ31B	40 570	330	40 730	250	40 250	230	40 850	110
No. 17...	Control (no couple)[c]	...	...	40 260	510	40 520	890	41 030	1330	40 350	520

[a] Based on average of 3 bars from 1 panel for specimen Nos. 1 to 16, and 16 bars from 4 panels for specimen No. 17; reported as pounds per square inch.

[b] Chromium-plated type 304 stainless steel.

[c] Initial tensile properties based on average of 12 bars from 4 unexposed AZ31B-H24 panels (0.188 in. thickness): 19.5 per cent elongation and 40,500 psi tensile strength.

TABLE X.—TENSILE STRENGTH[a] OF ZK60A-T5 PANELS AFTER 1 YEAR'S EXPOSURE AT FOUR SITES.

Specimen	Metal Coupled to ZK60A			New York		State College		Kure Beach 80-ft Site		Kure Beach 800-ft Site	
	Fastener	Strip A	Strip B	Avg	Range	Avg	Range	Avg	Range	Avg	Range
No. 1	6061	6061	99.99 per cent aluminum	50 710	1060	50 750	640	50 470	1 030	50 960	700
No. 2	6061	6061	99.99 per cent aluminum + 2 per cent magnesium	50 725	840	50 020	1040	50 310	1 090	51 095	680
No. 3	6061	6061	5356	50 300	990	50 820	690	50 310	890	50 760	1790
No. 4	6061	6061	6061	50 560	1240	50 570	740	50 310	600	51 130	920
No. 5	6061	6061	5052	51 130	610	50 860	840	50 595	1 240	51 220	850
No. 6	6061	6061	Alclad 7075	50 070	1380	51 140	870	49 110	1 290	51 040	1480
No. 7	Mild steel	Mild steel	Mild steel	48 670	840	49 095	1310	39 700	8 750	46 070	2660
No. 8	Brass	Brass	Brass	49 690	890	50 410	1100	31 075	11 630	47 855	2280
No. 9	304 stainless steel	304 stainless steel	304 stainless steel	49 830	770	50 570	1140	39 470	11 850	48 340	2920
No. 10	Plated 304 stainless steel[b]	Plated 304 stainless steel[b]	Plated 304 stainless steel[b]	50 300	750	50 840	650	48 570	1 590	49 940	7060
No. 11	Monel	Monel	Monel	49 800	750	50 350	1190	36 370	1 850	45 215	5830
No. 12	5056 rivets	5356	5356	50 370	940	50 680	990	49 920	2 120	50 660	640
No. 13	5056 rivets	5356	Alcan B54S	50 760	1070	50 820	540	49 970	1 430	50 540	1080
No. 14	6061	6061	ZK60A	50 500	1000	51 000	1020	50 590	1 500	51 200	930
No. 15	Plastic	ZK60A	ZK60A	50 660	840	50 815	1380	50 400	2 190	51 160	680
No. 16	5056 rivets	5356	ZK60A	50 790	1050	51 090	980	50 830	1 090	50 970	540
No. 17	Control (no couple)[c]	...	...	50 670	1440	51 050	1160	50 710	1 740	51 210	2830

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[a] Based on average of 6 bars from 2 panels for specimens Nos. 1 to 16, and 16 bars from 4 panels for specimen No. 17; reported as pounds per square inch.

[b] Chromium-plated type 304 stainless steel.

[c] Initial tensile properties based on average of 12 bars from 4 unexposed ZK60A-T5 panels (0.202 in. thickness): 14.5 per cent elongation in 2 in. and 49,900 psi tensile strength.

in ASTM Methods E 8. The difference between the tensile strength of the control panels and the tensile strength of the panels to which various metals were coupled was considered a measure of the severity of the galvanic attack suffered by the magnesium panels.

TABLE XI.—PER CENT LOSS IN TENSILE STRENGTH RESULTING FROM GALVANIC CORROSION OF AZ31B-H24 DURING 1 YEAR'S ATMOSPHERIC EXPOSURE.[a]

Specimen	Metal Coupled to AZ31B			Exposure Site			
						Kure Beach	
	Fastener	Strip A	Strip B	New York	State College	80-ft Site	800-ft site
No. 15...	Plastic	AZ31B	AZ31B	+0.2	2.3	0.1	+0.4
No. 16...	5056 Rivets	5356	AZ31B	+0.8	+0.5	1.9	+1.2
No. 14...	6061	6061	AZ31B	1.2	+2.1	2.3	+0.3
No. 1....	6061	6061	99.99 per cent aluminum	0.4	0.9	1.6	1.1
No. 2....	6061	6061	99.99 per cent aluminum + 2 per cent magnesium	+0.6	1.0	2.4	+0.4
No. 5....	6061	6061	5052	0.6	+0.5	2.9	0.1
No. 13...	5056 Rivets	5356	Alcan B54S	2.1	1.5	3.4	+0.9
No. 12...	5056 Rivets	5356	5356	1.3	0.3	2.9	+1.1
No. 3....	6061	6061	5356	0.5	0.2	2.6	+0.8
No. 4....	6061	6061	6061	0.2	+0.2	2.2	+0.6
No. 6....	6061	6061	Alclad 7075	1.4	+0.5	6.2	0.6
No. 9....	304 stainless steel	304 stainless steel	304 stainless steel	2.8	0.9	10.0	4.7
No. 10...	Plated 304 stainless steel[b]	Plated 304 stainless steel[b]	Plated 304 stainless steel[b]	0.4	+0.1	10.8	0.7
No. 11...	Monel	Monel	Monel	3.8	1.1	11.9	3.9
No. 7....	Mild steel	Mild steel	Mild steel	6.1	1.5	13.3	5.2
No. 8....	Brass	Brass	Brass	6.5	1.8	16.1	6.0

[a] Per cent loss in tensile strength is based on difference between average tensile strength of uncoupled AZ31B control panels (Item 17) and AZ31B panels coupled to various metals.

[b] Chromium-plated type 304 stainless steel.

Test Results:

The relative effect of the various dissimilar metals on the galvanic corrosion of AZ31B and ZK60A, as determined from visual inspection of cleaned panels, is presented in Tables VII and VIII, respectively. Although the difference between metals adjacent to each other in Tables VII and VIII are small, an attempt was made to rate these metals according to increasing galvanic effect within each rating group. The ratings are also comparable for AZ31B and ZK60A and for each of the four exposure sites.

In general, the galvanic attack tended to be slightly greater near the lower faying edge of strip *B*. There was no significant difference between the degree of attack on the skyward and groundward side of the magnesium panels. The galvanic attack was also limited to within about $\frac{1}{8}$ in. from the faying edge of strip *B* (see Figs. 3 and 4).

The average tensile strength and range in tensile strength of tension specimens machined from the AZ31B and ZK60A panels after one year's exposure in four different environments are summarized in Tables IX and X, respectively. The corresponding percentage loss in tensile strength is presented in Tables XI and XII.

Discussion of Results:

Visual examination indicated more distinct differences in the galvanic effect of the various dissimilar metals coupled to AZ31B and ZK60A than did the per cent loss in tensile strength data. However, both methods of evaluation rated the relative galvanic effect of the dissimilar metals in approximately the same order when coupled to either AZ31B or ZK60A.

TABLE XII.—PER CENT LOSS IN TENSILE STRENGTH RESULTING FROM GALVANIC CORROSION OF ZK60A-T5 DURING 1 YEAR'S ATMOSPHERIC EXPOSURE.[a]

Specimen	Metal Coupled to ZK60A			Exposure Site			
				New York	State College	Kure Beach	
	Fastener	Strip A	Strip B			80-ft Site	800-ft Site
No. 15...	Plastic	ZK60A	ZK60A	0.0	0.5	0.6	0.1
No. 16...	5056 rivets	5356	ZK60A	+0.2	+0.1	+0.2	0.5
No. 14...	6061	6061	ZK60A	0.3	0.1	0.2	0.0
No. 1....	6061	6061	99.99 per cent aluminum	+0.1	0.6	0.5	0.5
No. 2....	6061	6061	99.99 per cent aluminum + 2 per cent magnesium	+0.1	2.0	0.8	0.2
No. 5....	6061	6061	5052	+0.9	0.4	0.2	0.0
No. 13...	5056 rivets	5356	Alcan B54S	+0.2	0.4	1.5	1.3
No. 12...	5056 rivets	5356	5356	0.6	0.7	1.6	1.1
No. 3....	6061	6061	5356	0.7	0.4	0.8	0.9
No. 4....	6061	6061	6061	0.2	0.9	0.8	0.1
No. 6....	6061	6061	Alclad 7075	1.2	+0.2	3.2	0.3
No. 9....	304 stainless steel	304 stainless steel	304 stainless steel	1.7	0.9	22.2	5.6
No. 10...	Plated 304 stainless steel[b]	Plated 304 stainless steel[b]	Plated 304 stainless steel[b]	0.7	0.4	4.2	2.5
No. 11..	Monel	Monel	Monel	1.7	1.4	28.3	11.7
No. 7....	Mild steel	Mild steel	Mild steel	3.9	3.8	21.7	10.0
No. 8....	Brass	Brass	Brass	1.9	1.2	38.7	6.5

[a] Per cent loss in tensile strength based on difference between average tensile strength of uncoupled ZK60A control panels (Item 17) and ZK60A panels coupled to various metals.

[b] Chromium-plated type 304 stainless steel.

The tensile data revealed a greater number of anomalies, but these, as well as the small differences in tensile strength loss, can be attributed to the normal scatter in tensile strength data resulting from variation in the tensile properties of the magnesium panels and the accuracy of tension testing.

The following additional observations are also based on the visual ratings presented in Tables VII and VIII and the per cent loss in tensile strength data summarized in Tables XI and XII:

1. The seven aluminum alloys caused the least galvanic corrosion when coupled to either AZ31B or ZK60A.

2. All aluminum alloys were approximately equivalent in their galvanic effect on the magnesium alloys, with Alclad 7075 being slightly more severe than the other aluminum alloys.

3. There was no significant difference between the galvanic corrosion caused by high-purity and commercial-purity aluminum alloys. (The iron content of the aluminum alloy strips varied from 0.004 to 0.23 per cent.)

4. The magnesium alloys suffered the severest galvanic corrosion when in con-

tact with brass, monel, mild steel, and stainless steel.

5. Chromium plating on stainless steel was effective in reducing the galvanic corrosion caused by stainless steel alone.

6. Variation in the composition of strip *A* had no significant effect when either AZ31B or ZK60A was coupled to itself (specimen Nos. 14, 15, 16).

7. The magnesium alloys suffered the greatest amount of galvanic corrosion in the severe marine environment at the Kure Beach 80-ft site. The milder marine atmosphere at the Kure Beach 800-ft site was only slightly more corrosive than the New York Area and State College locations, which were the least corrosive.

8. Alloy ZK60A generally underwent slightly more galvanic attack than did AZ31B under comparable conditions of exposure.

Laboratory tests[6] have shown that high-purity aluminum causes less galvanic attack to magnesium alloy AZ31B than commercial-purity aluminum alloys when exposed in a 3 per cent NaCl solution. These tests also revealed that the presence of magnesium in aluminum alloys, or magnesium ion in the environment, can greatly suppress the harmful effect of iron, copper, and nickel impurities in commercial-purity aluminum alloys. Thus, this may explain why no significant difference was observed, especially on the panels exposed at the Kure Beach 80-ft site, in the galvanic attack caused by the high-purity and commercial-purity aluminum alloys. It is also very possible that the environment at the other exposure sites was not sufficiently aggressive to show up a significant difference among the various aluminum alloys.

The results of this atmospheric exposure study are consistent with similar results obtained in Part I[1] (disk-type couples) and Part II[2] (spool and wire couples) of the magnesium galvanic couple program sponsored by Subcommittee VIII in revealing that magnesium alloys can be coupled to most aluminum alloys without suffering significant galvanic attack. The data also indicate that certain precautions, such as the use of organic coatings, insulating tapes, or shims of more compatible materials between the principal components of the couple, should be taken whenever it is necessary to couple magnesium alloys with brass, monel, or steel alloys.

The type of panel being used in this program is effective in showing the relative galvanic effect of various dissimilar metals when in contact with magnesium alloys and exposed in different outdoor environments. Longer exposures will probably reveal greater differences between the tensile strength of the uncoupled control panels and the coupled panels, as well as in the galvanic ratings of the various dissimilar metals when coupled to magnesium alloys.

Acknowledgments:

Materials and assistance in the fabricating, assembling, and processing of the panels for this program were furnished by the following companies: Aluminum Company of America, Aluminium Limited, The American Brass Co., Bell Telephone Laboratories, Inc., Bohn Aluminum & Brass Corp., The Dow Chemical Co., The International Nickel Co., and The U. S. Steel Corp.

Respectfully submitted on behalf of the subcommittee,

J. J. CASEY

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[6] M. R. Bothwell, "Galvanic Relationships Between Aluminum Alloys and Magnesium Alloys," *Journal*, Electrochemical Society, Vol. 106, p. 1014 (1959).

REPORT OF COMMITTEE B-4

ON

METALLIC MATERIALS FOR THERMOSTATS AND FOR ELECTRICAL RESISTANCE, HEATING, AND CONTACTS*

Committee B-4 on Metallic Materials for Thermostats and for Electrical Resistance, Heating and Contacts held three meetings during the year: in Atlantic City, N. J., on June 28, 1960; at Skytop Lodge, Skytop, Pa., on November 2, 1960; and in Washington, D. C., on February 15, 1961.

The committee consists of 81 members, of whom 27 are classified as producers, 39 as consumers, and 15 as general interest members.

At the February meeting, A. L. VanEmden was elected to Honorary Membership in Committee B-4 for his outstanding services and contributions to Subcommittee IV. Mr. VanEmden has recently retired from active service with the Bureau of Ships. The other Honorary Members of this committee are P. H. Brace, F. E. Carter, Ragnar Holm, R. D. Van Nordstrand, and G. F. Geiger.

As it has done in the past, Committee B-4 again cooperated with The Pennsylvania State University in an Engineering Seminar on Electrical Contacts. The Seminar was held from June 11 to 16, 1961.

The committee is also cooperating with the University of Maine in the preparation of a program for the International Symposium on Contacts, which will be held at the University of Maine, Orono, Maine on November 14 to 16, 1961.

The committee continues to sponsor the Bibliography and Abstracts on Electrical Contacts published as *ASTM STP 56G* and annual supplements. In 1960, the Society published Supplement *ASTM STP 56N* covering the literature and abstracts for 1959.

Subcommittee III is preparing a bibliography on thermostat bimetals which is expected to be available within the next year. This bibliography will serve a similar purpose and have a similar format to that of the ASTM contact bibliography.

During the past year, Subcommittee IV on Contact Materials has had a special speaker at two of its meetings. At the Annual Meeting in Atlantic City, L. K. Jones of the Sandia Corp. gave an illustrated talk on "Materials and Process Variables and Their Effect on Contact Resistance." At the Skytop meeting, A. Keil of The Dr. E. Dürrwächter Co., Pforzheim, Germany, gave an illustrated talk on "Some Observations on Materials for Electrical Contacts."

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1960 Annual Meeting, Committee B-4 presented to the Society through the Administrative Committee on Standards the new Tentative Method of Test for Mechanical

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

Torque Rate of Spiral Coils of Thermostat Metal (B 362 - 60 T). The method was accepted by the Standards Committee on September 29, 1960, and it appears in the 1960 Supplement to Book of ASTM Standards, Part 3.

PROPOSED DEFINITIONS TO BE PUBLISHED AS INFORMATION

The committee recommends for publication as information only the Definitions of Terms Relating to Electrical Contacts and Their Use as appended hereto.[1]

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Method of Test for Surety of Make of Electrical Contact Materials (**B 340 - 59 T**)[2] be approved for reference to letter ballot of the Society for adoption as standard without revision.

ADOPTION OF TENTATIVE AS STANDARD WITH REVISIONS

The committee recommends that the Tentative Method of Test for Resistance Characteristics of Microcontacts (**B 326 - 58 T**)[3] be approved for reference to letter ballot of the Society for adoption as standard with revisions as appended hereto.[4]

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for more than six years without revision:

Standard Method of Test for:

Resistivity of Metallically Conducting Resistance and Contact Materials (**B 63 - 49**).
Thermoelectric Power of Electrical Resistance Alloys (**B 77 - 33**),
Temperature-Resistance Constants of Alloy Wires for Precision Resistors (**B 84 - 52**),
Temperature-Resistance Constants of Sheet-Materials for Shunts and Precision Resistors (**B 114 - 45**),
Effect of Controlled Atmospheres Upon Alloys in Electric Furnaces (**B 181 - 50**),
Life Test of Electrical Contact Materials (**B 182 - 49**), and
Hardness of Electrical Contact Materials (**B 277 - 55**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Electrical Heating and Resistance Materials (C. W. Armstrong, chairman) has been reviewing the specifications which come under its jurisdiction and certain changes will be recommended.

Subcommittee I has taken over jurisdiction from Subcommittee II of B 181 - 50, Standard Method of Test for Effect of Control Atmospheres Upon Alloys in Electric Furnaces. It is being retained in Committee B-4 even though the other activities of Subcommittee II have been transferred to Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys. Subcommittee I will review shortly the Tentative Method of Accelerated Life Test of Iron-Chromium-Aluminum Alloys for Electrical Heating (B 78 - 59 T).

A specific effort is being made to interest consumers in further work on Specification B 267 - 60 T for Wire for Use in Wirewound Resistors.

Subcommittee II on Wrought and Cast Alloys for Structural Use at High Temperatures has been discharged. The activities, except those noted above, have been taken over by ASTM Committee A-10.

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[1] See p. 232.

[2] 1959 Supplement to Book of ASTM Standards, Part 3.

[3] 1958 Book of ASTM Standards, Part 3.

[4] The revised standard appears in the 1961 Book of ASTM Standards, Part 3.

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee III on Thermostat Metals (U. U. Savolainen, chairman) is investigating additional properties of thermostat bimetals which now include flexivity, thermal emissivity, thermal conductivity, corrosion, thermal deflection rate of coils, and bond strength. In addition, a bibliography on thermostat metals is in preparation which will cover all of the important publications in this field.

Subcommittee IV on Contact Materials (J. D. Kleis, chairman) is continuing to promote the applicability of a test machine as described by Specification B 182 - 49 to include tests on the welding characteristics of contact materials, especially on contact bounce and its effect on welding.

The subcommittee prepared for criticism the proposed Definitions of Terms Relating to Electric Contacts and Their Use, appended hereto.[1]

Work is continuing on high current contacts to evaluate test procedures for measuring the erosion rates of refractory materials under arcing circumstances, and on microcontacts with an investigation of the characteristics of the machine described in Tentative Method B 326 - 58 T and the contact bounce which is developed on this machine.

Atmospheric exposure tests with crossed wire contacts are in progress at the ASTM test sites at Newark, N. J., and Kure Beach, N. C. Efforts are being made to expand this activity, and interested companies are being solicited for funds.

In the field of sliding contacts, it has been decided that this work will be limited initially to metal to metal contacts and arrangements are being made to test under controlled humidity circumstances. In addition, this work will investigate various lubricating materials.

This report has been submitted to letter ballot of the committee, which consists of 81 members; 63 members returned their ballots, of whom 58 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

ERLE I. SHOBERT II,
Chairman.

C. K. STROBEL,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee B-4 presented to the Society through the Administrative Committee on Standards the recommendation that the Standard Method of Test for Temperature-Resistance Constants of Alloy Wires for Precision Resistors (B 84 - 52) be revised and reverted to tentative. This recommendation was accepted by the Standards Committee on September 12, 1961, and the revised tentative method appears in the 1961 Book of ASTM Standards, Part 3.

APPENDIX I

PROPOSED DEFINITIONS OF TERMS RELATING TO ELECTRIC CONTACTS AND THEIR USE[1, 2]

These are proposed definitions and are published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

INTRODUCTION

The terms included in this list are those that are peculiar to electric contacts or general terms that have a specific meaning when related to electric contacts. The definitions were prepared assuming that the reader has a general knowledge in a physical science but is unfamiliar with the terminology of the literature of electric contacts.

A-Spot.—The conducting areas of two mating contacts through which current flows from one contact to the other.

Activation.—The contamination of the metal surface of a contact that gives rise to greater arcing (during either the "making" or "breaking" of an electrical circuit) than would occur at clean surfaces. This phenomenon is produced by repeated operation of a pair of current-carrying contacts in the presence of organic vapor.

Ambient Atmosphere.—The atmosphere surrounding the contacts.

Anode Arc.—The arc that occurs when the contact spacing is less than the critical value that depends on the current and contact material. The anode spot is small and heated to a higher temperature than the cathode spot. Material transfer is from the anode to the cathode.

Anodic Transfer.—Material transfer from the anode.

Arc.—A self-sustaining electric discharge characterized by a high cathode temperature, low cathode voltage drop, and high current density.

Arc Transfer.—Material transfer that occurs due to the action of an arc.

Blowout.—The displacement and lengthening of an arc to facilitate extinction. This blowout effect can be achieved by magnetic field, air blast, etc.

Bridge Transfer.—Material transfer that occurs without the presence of an electrical discharge. The bridge of molten contact material that connects the two separating contacts does not rupture in the middle, thus there is a gain of material on one contact and a loss of material from the other.

Brush Force.—See *Contact Force.*

Butting Contacts.—Contacts that close without sliding or rolling action but move in a direction perpendicular to the contact faces.

Carbonaceous Deposits.—Particles of carbon, or of material of high carbon content, that are the result of decomposition of organic matter by an electrical discharge or are generated external to the contact

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[1] These proposed definitions are under the jurisdiction of the ASTM Committee B-4 on Metallic Materials for Thermostats and for Electrical Resistance, Heating, and Contacts.

[2] Published as information only, June, 1961.

system and arrive at the contact as a contaminant.

Cathode Arc.—The arc that occurs when the contact spacing exceeds the critical value that depends on the current and contact material. The cathode spot is smaller than the anode spot and thus is heated to a higher temperature. Material transfer is from the cathode to the anode.

Cathodic Transfer.—Material transfer from the cathode.

Constriction Resistance.—The portion of contact resistance that results from the lines of current being bent and constricted because the current can flow from one contact to the other only through the a-spots.

Contact Closing Force.—The dynamic force between the contacts during closure.

Contact Force.—The force holding contacts together.

Contact Pressure.—The force per unit area of physical contact. This term is frequently used improperly when contact force is meant. The area of physical contact is difficult to determine and is usually quite different from the apparent area of contact.

Contact Resistance.—The resistance offered to the flow of current by a pair of closed contacts.

Contaminant.—A foreign material present on, or in, the contact surface.

Deformation, Mechanical.—A change in shape produced by mechanical means.

Delamination.—Separation at the interface of a composite material. Also used to describe a laminar separation in a homogeneous material due to a fault.

Erosion, Electrical.—The loss of contact material due to action of an electrical discharge.

Film.—An adherent layer of a foreign substance on the contact surface.

Fine Transfer.—See *Bridge Transfer.*

Fritting (A-Fritting).—An electric breakdown between mating metallic contacts, separated by an insulating film, which occurs when the field strength exceeds approximately 1,000,000 v per cm and results in metallic bridges through the film.

Fritting Voltage.—The value of voltage at which fritting occurs.

Glow Discharge.—A self-sustaining electric discharge characterized by a relatively low cathode temperature, high cathode voltage drop, and low current density, commonly occurring under conditions of relatively low pressure.

High Resistance.—Contact resistance exceeding an arbitrary specified limit.

Inrush Current.—A transient current that exists at the instant of contact closure and persists for a relatively short time.

Load Current.—The number of amperes carried in an electrical circuit.

Lock.—See *Mechanical Interlock.*

Material Transfer.—The carry-over of material from one contact to the other of a mating pair of contacts.

Mechanical Deformation.—See *Deformation, Mechanical.*

Mechanical Interlock.—The condition where contacts do not separate because of the interference of surface asperities.

Mechanical Wear.—See *Wear, Mechanical.*

Metal Transfer.—See *Material Transfer.*

Needle Transfer.—Material transfer that results in a build-up with a small diameter and a relatively great length.

Negative Transfer.—Material transfer that results in build-up on the negative contact. Care should be used not to confuse this term with *Cathodic Transfer.*

Opening Force.—The force available to open the contacts.

Pitted Contact.—A contact that has numerous discrete hollows in its surface.

Plasma.—A luminous highly ionized column between cathodic and anodic regions characterized by relatively small space charges—about an equal number of positive and negative carriers per cubic centimeter.

Positive Transfer.—Material transfer that results in a build-up on the positive contact. Care should be used not to confuse this term with *Anodic Transfer.*

Shortest Arc.—An arc with no plasma, or the plasma is so short that its voltage drop is negligible. No arc can burn with a length that is shorter than the thickness of the cathode layer.

Showering.—Sometimes used to describe transient electric discharges.

Sliding Contacts.—Contacts where the motion of one contact is parallel to the surface of the mating contact.

Spark.—A short-lived electric discharge that may be an arc, glow discharge, or a discharge in the state of development before equilibrium.

Static Contacts.—An electrical junction connected or separated infrequently.

Steady-State Current.—The value of current that exists essentially unchanged for long periods of time.

Stick.—A failure of contacts to separate.

Sulfide.—The result of the chemical reaction of the contact material with sulfur. See also *Tarnish.*

Supply Voltage.—The potential available at the source of electric current.

Tarnish.—The chemical compound on the surface of a contact resulting from the reaction of the contact material and the surrounding atmosphere.

Wear, Mechanical.—The loss of contact material due to mechanical action.

Weld.—A metallic bond between mating contacts as a result of electric heating and contact pressure.

Wiping Contacts.—Contacts that have some sliding motion during opening or closing.

REPORT OF COMMITTEE B-5

ON

COPPER AND COPPER ALLOYS, CAST AND WROUGHT*

Committe B-5 on Copper and Copper Alloys, Cast and Wrought, held two meetings during the year: at Niagara Falls, Ont., Canada, in September, 1960, and at New York City in January, 1961. At both of these sessions the Executive Subcommittee, Subcommittees W-1, W-2, W-3, W-4, F-1, G-3, and several task groups met. At the January session, the Supervisory Group to ASA on ISO/TC-26 also met.

The committee consists of 131 members of whom 104 are voting members; 43 are classified as producers, 37 as consumers; and 24 as general interest members.

The committee regrets to report the death on March 25, 1961, of Sidney Rolle, who was active in the committee for many years, was chairman of the Joint Committee on Classification of Coppers from its establishment until November, 1960, and was a member of the Supervisory Group to ASA on ISO/TC 26.

Appointments within the committee were: W. D. France, First Vice-Chairman, as Membership Chairman, M. L. Steinbuch as Vice-Chairman of Subcommittee F-1, on Cast Products, and W. E. Sartor as Secretary of Subcommittee W-4.

Appointments of representatives to other ASTM committees were: W. J. Anderson to Committee E-3 on Chemical Analysis of Metals; K. B. Bissell to Committee E-1, Task Group C on Thermal Conductivity for Metals; E. J. Hagan to Committee E-10 on Radioisotopes and Radiation Effects; E. H. Kinelski to Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys; W. E. Sartor to Committee E-7 on Nondestructive Testing and to Committee E-11 on Quality Control of Materials; and Stephen Teleshak to Committee E-1, Subcommittee 30 on Bend Test for Ductility. Other representatives appointed were: W. D. France to ASTM Division of Materials Sciences; M. F. W. Heberlein to ASA Sectional Committee C-7 on Bare Electrical Conductors; and J. M. Keyes to SAE Non-Ferrous Metals Technical Committee.

At the September meeting, a paper entitled "The Tensile Properties of Hard-Drawn Alloy A Phosphor Bronze Rod,"[1] by G. R. Gohn and W. G. Bader of Bell Telephone Laboratories, was presented by G. R. Gohn. At the January meeting the following two papers were presented: "Pressure Differential Testing of Tubing,"[2] by G. H. Symons of Wolverine Tube Division of Calumet & Hecla, Inc.; and "Properties of 60-40 Copper-Nickel Alloy at Temperatures Ranging Up to 1050 F"[3], by W. F. Simmons of Battelle Memorial Institute and B. J. Sirois of Phelps Dodge Copper Products Corp. An added highlight of the September meeting was a social hour sponsored by the new representatives of the Canadian Standards Assn. to Committee B-5.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] Published in *Proceedings*, Am. Soc. Testing Mats., Vol. 60, p. 843 (1960).

[2] To be published in *Materials Research & Standards*.

[3] See p. 247.

Recommendation Accepted by the Administrative Committee on Standards

Subsequent to the 1960 Annual Meeting, Committee B-5 presented to the Society through the Administrative Committee on Standards a recommendation for revision and reversion to tentative of the Standard Specification for Bronze Castings for Bridges and Turntables (**B 22 - 52**). This recommendation was accepted by the Standards committee on December 5, 1960, and the revised specification was published as a separate reprint. As noted under activities of Subcommittee F-1, Specification B 22 again is being adopted as standard.

Recommendations Affecting Standards

The committee is recommending 4 new specifications for publication—3 as standards for immediate adoption and 1 as tentative; the adoption as standard of 3 tentatives—1 without revision and 2 with revision; the adoption as standard of 1 tentative revision; the revision of 21 standards for immediate adoption; the reapproval of 16 standards; and the withdrawal of 1 standard.

The standards and tentatives affected, together with the revisions recommended, are covered in detail in the Appendix to this report.[4]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

Activities of Subcommittees

Executive Subcommittee (W. H. Jennings, chairman).—Seven replacement liaison appointments were arranged; and new liaison appointments were arranged to Committee E-1, Task Group C on Thermal Conductivity Test for Metals, Committee E-10 on Radioisotopes and Radiation Effects, and the ASTM Division of Material Sciences. It was decided not to establish liaison with Committees E-15 on Industrial Chemicals and E-19 on Gas Chromatography.

Action was taken to establish Joint Task Groups for Surface Oxidation of Oxygen-Free Copper and for Specifying Lengths.

ASTM Headquarters was requested to prepare a 1960 edition of the Compilation of Standards on Copper and Copper Alloys as sponsored by Committee B-5.

Other items considered by this subcommittee included industry needs for ASTM specifications, use of non-voting proxies, and work of the Joint Committee on the Classification of Coppers.

Work under way or under consideration comprises a study of packing and marking requirements, the preparation of specifications on fasteners, a study of methods for indicating revisions to specifications, the recommendation of specifications for adoption by ASA, and the heat treatment requirements in specifications adopted by the ASME Boiler and Pressure Vessel Committee.

Subcommittee W-1 on Plate, Sheet and Strip (A. E. Moredock, chairman) submitted a new specification on Copper Sheet for Building Construction. Revisions were recommended to add length and width tolerances to Specifications B 11, B 96, and B 100; to incorporate new CABRA straightness tolerances in specifications B 19, B 130, B 248, and B 291; to revise Table II and to delete Rockwell F scale values in specification B 36; and to incorporate revisions and to delete

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[4] See p. 240.

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Rockwell G values from Table I of specification B 103. Reapproval was recommended for standard specifications B 129, B 131, and B 169.

Work under way includes (1) the development of new specifications for: copper foil, sheet and strip for printed circuits; strip for flexible hose; copper-nickel-silicon sheet and strip; and copper-alloy clad steel plate, with liaison from Committee A-1, Subcommittee XI; and (2) the revision of specifications: B 36, B 122, and B 131 to add limits on impurities; B 100 as regards mechanical requirements; B 103 to modernize hardness values; B 122 to modernize hardness requirements, and to add 90/10 copper-nickel alloy and requirements for annealed copper-nickel alloys; B 194 for general improvements; and B 248 as regards sections on sampling and retests. A joint study with Subcommittees W-2 and W-4 is being made on the surface oxidation of oxygen-free copper; and there is a study on non-uniformity of grain size in thin strip.

Subcommittee W-2 on Rod, Bars and Shapes (O. A. Klema, chairman), submitted a new specification on Copper-Zinc-Silicon Alloy Rod.

Revisions were recommended for specification B 12 to add length tolerances; for specification B 124 to change from "exact" to "specific" when specifying lengths; and for specification B 249 as required to cover the new standard Specification for Copper-Zinc-Silicon Alloy Rod. Reapproval was recommended for Standard Specification B 124.

Work under way includes (1) the development of new specifications on low-leaded brass rod; and copper-nickel-silicon alloy rod and bar; and (2) the revision of specifications: B 16, Tables I and II; B 21 and B 140, as regards tensile strength requirements; B 151 on hardness requirements; B 124 and B 283 for list of alloys; B 187 and B 249 on retest sections; B 196 for general improvement; and B 283 for mechanical properties. There is a joint study with Subcommittees W-1 and W-4 on the surface oxidation of oxygen-free copper.

Subcommittee W-3 on Wire and Wire Rod (M. J. Mianulli, chairman) has recommended revisions to change the tensile strength requirements on annealed alloy B in specification B 99; to add better references to specification B 250 on dimensional tolerances in specification B 197; and to specify minimum sizes of wire to which straightness tolerance apply in specifications B 250 and B 272. Adoption as standard was recommended for tentative specifications B 250 and B 272. Reapproval was recommended for Standard Specification B 134.

Work under way includes (1) the development of a new specification for copper-nickel-silicon alloy wire; and (2) the revisions of specifications: B 134 to add limits on impurities, B 134 and B 250 on requirements for rectangular and square wire, and B 197 for general improvements.

Subcommittee W-4 on Pipe and Tube (J. M. Keyes, chairman) submitted a new Specification for Seamless Rectangular Copper and Copper Alloy Wave Guide.

Revisions were recommended for the following specifications: B 88, to include dimensional tolerances and general requirements previously contained in B 251; B 75, B 135, B 188, and B 251 to add twist tolerances on square and rectangular tube; B 188, B 251, B 302, B 306, and B 315 to clarify squareness of cut; B 188 and B 251 as required by the rewriting of B 88; and B 302 to change from "nominal size" to "standard size" in several places. Withdrawal of Specification B 13 was recommended, since a survey has shown that this specification is no longer in general use.

Work under way includes (1) the development of new specifications on welded copper and copper alloy tube; and "U" bend copper and copper alloy heat exchanger and condenser tube; and (2) the revision of specifications: B 43, B 111, and B 135 to add limits on impurities; B 111 to add 60/40 copper-nickel alloy; B 280 to add 1 in. and 1⅛-in. type L sizes of water tube; and B 315 to add Alloy B of B 97. There is a joint study with Subcommittees W-1 and W-2 on the surface oxidation of oxygen-free copper, and also a study on microporosity in oxygen-free copper.

Subcommittee F-1 on Castings and Ingots for Remelting (G. H. Clamer, chairman) submitted a new Specification for Copper-Nickel Alloy Castings. Revisions were recommended for the following specifications: B 143 and B 145 to add requirements on impregnation of castings. Adoption as standard was recommended for Tentative Specification B 22. Reapproval was recommended for Standard Specifications B 52, B 53, B 66, B 67, B 132, B 144, B 146, B 147, B 148, B 149, B 198, and Classification B 119.

This subcommittee has worked out a program whereby ASTM specifications on copper alloy castings are used by the Manufacturers Standards Society, and all MSS material specifications have been discontinued.

Work under way includes efforts to promote the use of ASTM specifications on copper-alloy castings for Government purchases by means of suitable specification development; modification of chemical analysis sections of all Subcommittee F-1 specifications to include methods of spectrographic analysis; review of specifications B 30, B 145, B 271 and Classification B 119 for changes needed because of change in title of Specification B 62; and proposed change in wording of Classification B 119 as regards limit on zinc.

Subcommittee G-1 on Methods of Test (P. J. Strubhar, chairman) has under consideration the development of recommendations for revision of Table I of Specification B 103 and Table II of Specification B 122 to follow the pattern of the proposed revision of Table II of Specification B 36; and improvements in Methods E-8 to be recommended to Committee E-1.

Subcommittee G-2 on Tolerances (J. E. McGraw, chairman) continued to maintain excellent work relations with the Copper and Brass Research Assn. and received help from them on specific problems. The actions by Subcommittee W-1 on straightness tolerances and Subcommittee W-3 on minimum size of rectangular and square wire to which straightness tolerances apply were based on recommendations by Subcommittee G-2.

Subcommittee G-3 on Editorial and Publications (W. F. Roeser, chairman) gave considerable assistance to other subcommittees by continuing to review new and revised specifications for editorial consistency, and taking a prominent role in proposed revisions for the sampling and retest requirements of Specifications B 188 and B 249. Recommendations were made on the Proposed Recommendations on Form of ASTM Methods of Test.

Possible revisions are under consideration for sampling and retest sections of Specifications B 248, B 249, B 250, and B 251.

Supervisory Group to ASA on ISO/TC-26 (W. H. Jennings, chairman) approved the Draft Proposal ISO/TC-26 (Secretariat-39) 68 on Copper Wire Bars, Cakes, Slabs, Billets, Ingots and Ingot Bars. It was recommended that ASA participate in the work of TC/26 Subcommittee 1 on Chemical Analysis, with interest only in copper; and due to lack of USA interest, ASA consider discontinuance of participation in work of TC-26 Working Group 3 on Castings. Eight WG-1 first draft proposals on test methods and six WG-2 first draft proposals on classification of wrought products were voted upon.

Work under way includes planning for USA delegates to attend the meeting of ISO/TC-26 on June 20–24, 1961, at Koln/Rhein, Germany, planning for a related meeting of WG-4, and obtaining the help of Committee E-3 on Chemical Analysis of Metals in the development of ISO chemical analysis methods for coppers.

Floyd H. Wasser has been made a member of the Supervisory Group, replacing A. E. Beck, who resigned due to change of duties. John R. Freeman, Jr., has been added to the group and has been made Chairman of WG-4, in place of A. E. Beck.

This report has been submitted to letter ballot of the committee, which consists of 104 members; 96 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. H. JENNINGS,
Chairman.

G. C. MUTCH,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee B-5 presented to the Society through the Administrative Committee on Standards a tentative revision of the Standard Specification for Seamless Copper and Copper-Alloy Rectangular Wave Guide Tube (B 372 – 61). The tentative revision was accepted by the Standards Committee on November 7, 1961, and appears in the 1961 Book of ASTM Standards, Part 2.

APPENDIX I

RECOMMENDATIONS AFFECTING STANDARDS FOR COPPER AND COPPER ALLOYS, CAST AND WROUGHT

In this Appendix recommendations are given affecting certain specifications, both standard and tentative, referred to earlier in this report. The specifications appear in their present form in the 1958 Book of ASTM Standards, Part 2 or in the 1959 and 1960 Supplements to Book of ASTM Standards, Part 2; and in the 1960 edition of the B-5 Compilation.

NEW STANDARDS, IMMEDIATE ADOPTION

The committee recommends that the following specifications as appended hereto[1] be approved for reference to letter ballot of the Society for adoption as standards and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the specifications may be referred to letter ballot of the Society:

Standard Specifications for:

Copper Sheet and Strip for Building Construction,
Copper-Zinc-Silicon Alloy Rod, and
Seamless Copper and Copper-Alloy Rectangular Wave Guide Tube.

NEW TENTATIVE

The committee recommends for publication as tentative the Specification for Copper-Nickel Alloy Castings as appended hereto.[1]

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Specification for Bronze Castings for Bridges and Turntables (**B 22 – 60 T**) be approved for reference to letter ballot of the Society for adoption as standard without revision.

ADOPTION OF TENTATIVES AS STANDARD WITH REVISION

The committee recommends that the following tentative specifications be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

Tentative Specification for General Requirements for Wrought Copper-Alloy Wire (B 250 – 60 T):

Table IX.—Revise to conform to industry practice of CABRA Schedule FP-7, as shown in the accompanying Table I,[2] retaining the present table title and parenthetical references to ASTM designations.

Tentative Specification for Flat Copper Products with Finished (Rolled or Drawn) Edges (Flat Wire and Strip) (B 272 – 60 T):

Table VI.—Revise to conform to industry practice of CABRA Schedule FP-7 as shown in the accompanying Table I.[2]

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[1] The new standards and tentative appear in the 1961 Book of ASTM Standards, Part 2.

[2] The new tables are not included in this report but appear in the revised specification; see 1961 Book of ASTM Standards, Part 2.

ADOPTION OF TENTATIVE REVISIONS AS STANDARD WITH REVISION

The committee recommends that the tentative revisions of Section 1(*a*), Table I, and Table II of the Standard Specification for Phosphor Bronze Plate, Sheet, Strip, and Rolled Bar (**B 103 – 60**) approved for reference to letter ballot of the Society for adoption as standard with the following revision:

Table II.—Delete Rockwell G values from the revised Table II where they now appear, as they are not being used.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Specification for Copper Plates for Locomotive Fireboxes (B 11 – 58):

Section 12.—Change the heading to read "Dimensions, Weights, and Permissible Variations." Letter the present Section 12 as Paragraph *a* and add the following new Paragraphs (*b*), (*c*), and (*d*):

(*b*) Length tolerances for square sheared metal in all widths 120 in. and under for all lengths shall be plus and minus $\frac{1}{16}$ in. If tolerances are specified as all plus or all minus, double the values given.

(*c*) Length tolerances for sawed metal for all widths up to and including 120 in. shall be plus and minus $\frac{1}{8}$ in. If tolerances are specified as all plus or all minus, double the values given.

(*d*) Width tolerances for square sheared metal or sawed metal for widths up to and including 120 in. for all thickness, shall be plus and minus $\frac{1}{16}$in. If tolerances are specified as all plus or all minus, double the values given.

Standard Specification for Copper Rods for Locomotive Staybolts (B 12 – 58):

Section 12.—Delete the present Section 12 and Note 3 and replace with the following under the heading "Dimensional Tolerances:"

12. (*a*) For the purpose of determining conformance with the dimensional requirements prescribed in this specification, any measured value outside the specified limiting values for any dimension may be cause for rejection.

(*b*) *Diameter.*—The rods shall be round within 0.01 in., and shall not vary more than 0.01 in. over nor more than 0.005 in. under the specified size.

(*c*) *Length.*—The material shall be furnisdeh in stock lengths with ends, unless the order specifies stock lengths, specific lengths, or specific lengths with ends. Length tolerances and schedule of lengths shall be as prescribed in Tables I and II.

New Tables.—Add new Tables I and II as shown in the accompanying Tables II and III.[2]

Standard Specification for Cartridge Brass Sheet, Strip, Plate, Bar, and Disks (B 19 – 58):

Table XI.—Revise the straightness tolerances for slit metal and slit metal with rolled edges, to conform with industry practice of CABRA Schedule FP-4 as follows:

Width, in.	Straightness Tolerance, in.		
	As Slit Only		As Slit and Either Straightened or Edge Rolled
	Shipped in Rolls	Shipped Flat	Shipped Flat, in Rolls, or on Bucks
Over ¼ to ⅜, incl.	2	1½	½
Over ⅜ to ½, incl.	1½	1	½
Over ½ to 1, incl.	1	¾	½
Over 1 to 2, incl.	⅝	⅝	⅜
Over 2 to 4, incl.	½	½	⅜
Over 4	⅜	⅜	⅜

Standard Specification for Brass Plate, Sheet, Strip, and Rolled Bar (B 36 – 60):

Table II.—Revise Table II to conform to most recent practice, as shown in the accompanying Table IV.[2]

Standard Specification for Seamless Copper Tube (B 75 – 60):

Section 14.—Add a new Paragraph (*i*) to read: "(*i*) *Twist Tolerances for Rectangular and Square Tubes.*—See Section 8(*l*)."

Table V.—Change the last part of the heading of the first column to read: ". . . Distance Between Parallel Outside Surfaces, in."

Standard Specification for Seamless Copper Water Tube (B 88 – 58):

Revise as appended hereto.[3]

Standard Specification for Copper-Silicon Alloy Plate and Sheet for Pressure Vessels (B 96 – 58):

Section 11.—Change the heading to read "Dimensions, Weights, and Permissible Variations." Letter the present Section 11 as Paragraph *a* and add new Paragraphs *b*, *c*, and *d*, as shown above for Section 12 of Specifications B 11.

Standard Specifications for Copper-Silicon Alloy Wire for General Purposes (B 99 – 54):

Table II.—Under alloy B, change tensile strength of annealed temper from "43,000 to 55,000" to "38,000 to 55,000" psi.

Standard Specification for Rolled Copper-Alloy Bearing and Expansion Plates and Sheets for Bridge and Other Structural Uses (B 100 – 58):

Section 9.—Change the heading to read "Dimensions, Weights, and Permissible Variations." Letter the present Section 9 as Paragraph *a*, and add new Paragraphs (*b*), (*c*), and (*d*), as shown above for Section 12 of Specifications B 11.

Standard Specification for Commercial Bronze Strip (B 130 – 58):

Table VIII.—Revise the straightness tolerances for slit metal and slit metal with rolled edges to conform to industry practice of CABRA Schedule FP-4, as indicated above for Table XI of Specifications B 19.

Standard Specification for Seamless Brass Tube (B 135 – 60):

Section 11.—Add a new Paragraph (*i*) to read: "(*i*). *Twist Tolerances for Rectangular and Square Tubes.*—See Section 8(*l*)."

Table III.—In the heading of the second column, delete the word "Annealed."

Standard Specification for Tin Bronze and Leaded Tin Bronze Sand Castings (B 143 – 52):

Section 13.—Add a new Paragraph (*c*) to read as follows, relettering the present paragraph (*c*) and subsequent paragraphs accordingly:

> Impregnated castings may be employed where agreement is reached between producer and consumer covering impregnant procedure and marking.

Standard Specification for Leaded Red Brass and Leaded Semi-Red Brass Sand Castings (B 145 – 52):

Section 13.—Revise as indicated above for Section 13 of Specification B 143.

Standard Specification for Seamless Copper Bus Pipe and Tube (B 188 – 60):

Section 3.—Change the heading to read "Ordering Information."

Table III.—Change the heading of the first column to read "Standard Pipe Size, in."

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[3] The revised standard appears in the 1961 Book of ASTM Standards, Part 2.

Table IV.—Change the heading of the second column to read "Nominal Outside Diameter, in."

Change the major heading over Columns 5 through 10 to read "Nominal Wall Thickness and Tolerances, in."

Change heading over Columns 5, 7, and 9 from "Nominal" to "Wall Thickness."

Add a Footnote *b* referenced to heading "Tolerance" in Columns 6, 8, and 10. to read: "Maximum deviation at any one point."

Add a Footnote *c* referenced to the two type M tolerances for size ⅝-in. tube which are now blank to read: "The material is either not generally available or no tolerance has been established."

Add the following columns to Table IV under a new major heading "Cross-Sectional Area of Wall, sq in."

Standard Size, in.	Type K	Type L	Type M
⅜	0.069	0.051	0.037
½	0.089	0.074	0.053
⅝	0.108	0.093	...
¾	0.165	0.117	0.084
1	0.217	0.169	0.120
1¼	0.268	0.228	0.176
1½	0.351	0.295	0.292
2	0.533	0.452	0.377
2½	0.755	0.640	0.523
3	1.03	0.858	0.691
3½	1.32	1.11	0.924
4	1.68	1.39	1.20
5	2.50	1.96	1.72
6	3.58	2.63	2.70
8	6.69	4.98	4.25
10	10.4	7.76	6.60
12	14.9	10.4	9.47

Section 13.—Change Paragraph (*d*) to read ". . . the various standard sizes shall be as prescribed in Table III."

In Paragraph (*e*), change the word "nominal" in line 4 to read "standard."

Revise Paragraph (*j*) to read:

(*j*) *Length and Tolerances for Water Tube.*—The standard length and tolerances for copper water tube sizes shall be as follows:

TUBES FURNISHED IN STRAIGHT LENGTHS

Standard Size, in.	Type	Standard Length, ft	Tolerance, All Plus, in.
Up to 8, incl.	K, L, M	20	1
10	L, M	20	1
10	K	18	1
12	M	20	1
12	L	18	1
12	K	12	1

TUBES FURNISHED IN COILS

Standard Size, in.	Type	Standard Length, ft	Tolerance, All Plus, ft
Up to 1, incl.	K, L	60 and 100	2
1¼ and 1½	K, L	60	2
2	K, L	40	1

Change Paragraph (*l*) to read as follows:

(*l*) *Squareness of Cut.*—The departure from squareness of the end of any tube shall not exceed the following:

(*1*) *Round Tube or Pipe.*—For tube or pipe up to and including ⅝ in. in outside diameter, the departure from the square shall not exceed 0.010 in.; for tube or pipe over ⅝ in., the departure shall not exceed 0.016 in. per inch of diameter.

(*2*) *Square and Rectangular Tube.*—For tube up to and including ⅝ in. in dimension across the measured section the departure from the square shall not exceed 0.016 in.; for tube over ⅝ in., the departure shall not exceed 0.025 in. per inch of distance between outside parallel surfaces.

Add new Paragraph (*p*) to read as follows:

(*p*) *Twist Tolerances.—Rectangular and Square Tubes.*—The maximum twist about the longitudinal axis of drawn temper rectangular and square tubes shall not exceed 1 deg per foot of length, measured to the nearest degree, and the total angle of twist shall not exceed 20 deg when measured in accordance with the method described in Appendix I. The requirement is not applicable to tubes in the annealed temper or to tubes whose specified major dimension is less than ½ in.

Appendix I.—Add a new Appendix I to read as follows under the heading

"Method for Determining Twist in Rectangular and Square Tubes:"

*A*1. Measurements made to determine conformance to the twist requirements of Section 13(*p*) shall be as prescribed below.

*A*2. This horizontal method of measurement with a protractor segment is unaffected by edgewise or flatwise curvature of the tube, and permits measurement of the total angle of twist to the nearest degree (±0.5 deg).

*A*3. The angle of twist shall be determined as follows (see Fig. 1):

(*a*) The tube shall be placed on a flat horizontal reference plane. If the tube has noticeable flatwise curvature, it shall be positioned with the tube surface on the side of the convex curvature in contact with the reference plane. The surface at one end of the tube shall be held in intimate contact with the reference plane (Fig. 1 (*a*)) by suitable means, while the other end of the tube remains free.

(*b*) Using an engraved transparent protractor segment (Fig. 1 (*c*)), determine the angle line on the protractor which coincides to the nearest degree with the surface of the tube under consideration (Fig. 1(*b*)), while one end of the protractor segment contacts, or is kept parallel to the reference plane by means of parallel face blocks.

(*c*) The angle line so found represents the total angle of twist for the length of tube under consideration.

Figure 1.—Add a new Fig. 1 as shown in the accompanying Fig. 1.[4]

Standard Specification for General Requirements for Wrought Copper and Copper-Alloy Plate, Sheet, Strip, and Rolled Bar (B 248 – 59):

Tables XI and XII.—Revise the straightness tolerances to conform with industry practice of CABRA Schedule FP-4 as indicated above for Table XI of Specification B 19.

Standard Specification for General Requirements for Wrought Copper and Copper-Alloy Rod, Bar, and Shapes (B 249 – 60):

Add references to the new Standard Specification for Copper-Zinc-Silicon Alloy Rod (B 371 - 61) as follows:

Section 1.—Add title and designation number to present list.

Section 3(c).—Add designation number.

Section 4(b).—Add designation number opposite "225 g."

Section 8(b).—Add designation number to the paragraph starting "Table II."

Section 8(c).—Add designation number to the paragraphs starting "Table XII" and "Table XIV."

Tables II, XII, XIV and XV.—Add designation number under titles of Tables II, XII, XIV, and XV.

Appendix.—Add the designation number in the first column; in the second column, add the material "copper-zinc-silicon alloy"; in the third column, add the alloy designations "A" and "B"; and in the fourth column, add the density values 0.296 and 0.300 for "A" and "B," respectively.

Standard Specification for General Requirements for Wrought Seamless Copper and Copper-Alloy Pipe and Tube (B 251 – 60):

Section 1.—Delete B 88 from the list of specifications.

Section 4 (a).—In the paragraph headed "Portion Size," change the wording in the table of the last item in left-hand column to read: "0.2 per cent of total number of pieces in the lot, but not to exceed 10 sample pieces." In this same table, reference a new Footnote *a* to the heading of the second column, and add Footnote *a* to read: "[a] Each sample piece shall be taken from a separate tube."

Section 8.—Delete Paragraphs (*b*) (2), (*c*) (2), and (*d*) (2). Renumber present Paragraph (*d*) (3) as (*d*) (2), and delete "and Water Tube" from heading.

Delete Paragraph (*e*) (1). Renumber present Paragraph (*e*) (2) as (*e*) (1), and delete "and Water Tube" from heading.

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[4] The new figure is not included in this report but appears in the revised specification; see 1961 Book of ASTM Standards, Part 2.

Delete reference to B 88 from Paragraphs (*f*) and (*g*). Delete the last two sentences of Paragraph (*g*).

Delete Paragraph (*h*) (4).

Revise Paragraph (*i*) to read:

> (*i*) ***Squareness of Cut*** (*Applicable to ASTM Designations B 42, B 43, B 68, B 75, and B 135.*— The departure from squareness of the end of any tube shall not exceed the following:
>
> (*1*) *Round Tube or Pipe.*—For tube or pipe up to and including ⅝ in. in outside diameter, the departure from the square shall not exceed 0.010 in.; for tube or pipe over ⅝ in., the departure shall not exceed 0.016 in. per inch of diameter.
>
> (*2*) *Square and Rectangular Tube.*—For tube up to and including ⅝ in. in dimension across the measured section the departure from the square shall not exceed 0.016 in.; for tube over ⅝ in., the departure shall not exceed 0.025 in. per inch of distance between outside parallel surfaces.

Delete the words "copper water tube" from Paragraph (*j*) (1).

Add a new Paragraph (*l*) to read as follows:

> (*l*) *Twist Tolerances.—Rectangular and Square Tubes.*—The maximum twist about the longitudinal axis of drawn temper rectangular and square tubes applicable to ASTM Designations B 75 and B 135 shall not exceed 1 deg per foot of length, measured to the nearest degree, and the total angle of twist shall not exceed 20 deg when measured in accordance with the method described in Appendix III. The requirement is not applicable to tubes in the annealed temper or to tubes whose specified major dimension is less than ½ in.

Appendix II.—Delete "B 88" from Column 1.

Tolerance Tables.—Delete reference to Specification B 88 in Table VII. Delete the words "copper water tube" from Footnote *a* of Table X.

Tables II and VIII.—Delete these tables and renumber remaining tables, but reference a footnote to the heading "Tolerance Tables" to read:

> As a consequence of the incorporation in the revised Specification for Seamless Copper Water Tube (ASTM Designation: B 88) of the material formerly appearing in Table II on Standard Dimensions and Weights, and Tolerances in Diameter and Wall Thickness for Standard Copper Water Tube Sizes and Table VIII on Length Tolerances for Copper Tube in Coils, these tables were deleted from this Specification B 251 and subsequent tables were renumbered accordingly.

Appendix III.—Add a new Appendix III to read as shown above for Appendix I of Specification B 188, except in Section A1 refer to "Section 8(*l*)."

Figure 1.—Add a Fig. 1 as shown in the accompanying Fig. 1.[4]

Standard Specification for Copper-Zinc-Manganese Alloy (Manganese Brass) Sheet and Strip (B 291 – 60):

Tables XII and XIII.—Revise as indicated above for Table XI of Specification B 19.

Standard Specification for Threadless Copper Pipe (B 302 – 60):

Section 3(1).—Change "Nominal" to "Standard."

Table I.—Change "Nominal Pipe Size" to "Standard Pipe Size" in the first column.

Section 13.—Change "nominal sizes" to "standard sizes" in paragraph (*b*).

Change Paragraph (*f*) to read as follows:

> (*f*) *Squareness of Cut.*—The departure from squareness of the end of any pipe shall not exceed the following:

Pipe Size, Actual Outside Diameter, in.	
Up to ⅝ incl......	0.010 in.
Over ⅝..........	0.016 in. per in. of diameter

Standard Specification for Copper Drainage Tube (DWV) (B 306 – 59):

Section 11(f).—Change to read as follows:

> (*f*) *Squareness of Cut.*—The departure from squareness of the end of any tube shall not exceed the following:

Tube Size, Actual Outside Diameter, in.	
Up to ⅝, incl.....	0.010 in.
Over ⅝..........	0.016 in. per in. of diameter

Standard Specification for Copper-Silicon Alloy Seamless Pipe and Tube (B 315 – 59):

Section 13(f).—Revise as indicated above for Section 11(*f*) of Specification B 306.

WITHDRAWAL OF STANDARD

The committee recommends the withdrawal of the Standard Specification for Seamless Copper Boiler Tubes (**B 13 – 58**).

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Standard Specifications for:

Phosphor Copper (**B 52 – 48**),
Silicon Copper (**B 53 – 46**),
Bronze Castings in the Rough for Locomotive Wearing Parts (**B 66 – 52**),
Car and Tender Journal Bearings, Lined (**B 67 – 52**),
Copper and Copper-Alloy Forging Rod, Bar, and Shapes (**B 124 – 55**),
Cartridge Brass Cartridge Case Cups (**B 129 – 58**),
Commercial Bronze Bullet Jacket Cups (**B 131 – 55**),
Leaded High-Strength Yellow Brass (Manganese Bronze) Sand Castings (**B 132 – 52**),
Brass Wire (**B 134 – 52**),
High-Leaded Tin Bronze Sand Castings (**B 144 – 52**),
Leaded Yellow Brass Sand Casting for General Purposes (**B 146 – 52**),
High-Strength Yellow Brass (Manganese Bronze) and Leaded High-Strength Yellow Brass (Leaded Manganese Bronze) Sand Castings (**B 147 – 52**).
Aluminum Bronze Sand Castings (**B 148 – 52**)
Leaded Nickel Brass (Leaded Nickel Silver) and Leaded Nickel Bronze (Leaded Nickel Silver) Sand Castings (**B 149 – 52**),
Aluminum Bronze Plate, Sheet, Strip, and Rolled Bar (**B 169 – 55**), and
Silicon Bronze and Silicon Brass Sand Castings (**B 198 – 58**),

Standard Classification of:

Cast Copper-Base Alloys (**B 119 – 45**).

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following standards:

Standard Specification for Copper and Copper-Alloy Forging Rod, Bar, and Shapes (B 124 – 55):

Section *10(c)*.—Change the word "exact" to "specific" in the first paragraph.

Standard Specification for Copper-Beryllium Alloy Wire (B 197 – 52):

Section 9.—In Paragraph (*a*) change the word "table" to "tables" in the last line.

Change Paragraph (*b*) to read: "*Diameter.*—See Section 8(*b*) and Table II."

Add new Paragraphs (*c*), (*d*), (*e*), and (*f*) to read as follows:

(*c*) *Thickness.*—See Section 8(*c*) and Table IV.

(*d*) *Width.*—See Section 8(*d*), and Table VI.

(*e*) *Length* (*for flat wire only*).—See Section 8(*e*), Tables VII and VIII.

(*f*) *Straightness* (*flat wire*).—See Section 9 and Table IX.

APPENDIX II

PROPERTIES OF 60-40 COPPER-NICKEL ALLOY AT TEMPERATURES RANGING UP TO 1050 F*

By W. F. Simmons[1] and B. J. Sirois[2]

Synopsis

Some elevated-temperature properties for Cufenloy 40, a wrought alloy containing nominally 56 per cent copper, 41 per cent nickel, 2 per cent iron, and 1 per cent manganese, are given for both the annealed and drawn and stress-relieved temper. Physical properties reported include thermal expansion, thermal conductivity, electrical resistivity and conductivity, and dynamic modulus of elasticity. Mechanical properties include the short-time tensile properties and the long-time creep and creep-rupture properties for temperatures to 1050 F.

The alloy in both the annealed and in the drawn and stress-relieved temper exhibits a proper combination of strength properties, ductility, and stress-corrosion resistance to make its use advantageous in unfired pressure vessel applications at temperatures up to 900 F.

In 1959, Simmons, Sirois, Williams, and Jaffee (1)[3] reported on an extensive study of the properties obtainable in Cufenloy 30, a 70-30 copper-nickel alloy produced in a drawn and stress-relieved temper. This study had resulted from several requests from manufacturers of heat exchangers for copper-base-alloy tubes having improved mechanical properties at elevated temperatures. The data obtained in this investigation showed that heat exchanger tubes of this alloy could be made with sufficiently enhanced mechanical properties to warrant recognition by the ASME Code for Unfired Pressure Vessels of design factors approximately 50 per cent greater than those currently included in the Code for the annealed temper tubes of the same alloy.

The excellent resistance to corrosion of the copper-nickel family of alloys is amply substantiated by years of successful use in many applications, the most noteworthy being heat-transfer equipment such as marine and stationary power plants, and oil-refinery and chemical-processing plants.

The demand for alloys having excellent corrosion resistance combined with high mechanical properties led to the study of Cufenloy 30 initiated early in 1957. This was extended to include other copper-nickel alloys. Alloys containing nominally 65-35, 60-40, 55-45 and 50-50 copper-nickel, in which the iron content was varied in the range of 0.5 to 2.6 per cent, were investigated. The preliminary data obtained in these alloys showed that Cufenloy 40, a 60-40 copper-nickel alloy containing nominally 56 per cent copper, 41 per cent nickel, 2 per cent iron, and 1 per cent manganese did possess the desirably improved mechanical properties and lent itself to processing on standard mill equipment.

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* Presented at a meeting of Committee B-5 on Copper and Copper Alloys, Cast and Wrought, New York, N. Y., January 18, 1961.

[1] Division Chief, Battelle Memorial Inst., Columbus, Ohio.

[2] Director of Research, Phelps Dodge Copper Products Corp., Elizabeth, N. J.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

TABLE I.—MINIMUM MECHANICAL PROPERTIES FOR DUCTILITY AT ROOM TEMPERATURE.

	Temper	
	Annealed	Drawn, Stress Relieved
Tensile strength, min, psi...	64 000	85 000
Yield strength,[a] min, psi...	28 000	65 000
Elongation in 2 in., min, per cent:		
For wall thicknesses up to 0.048 in. incl.........	30	12
For wall thicknesses over 0.048 in..............	30	15
Minimum expansion of tube inside diameter with tapered pin, per cent...	30	20
Average grain size diameter, mm..............	0.010 to 0.045	...

[a] At 0.5 per cent elongation under load.

In addition to corrosion resistance and high strength properties, tubes intended for use in heat exchangers must be sufficiently ductile to permit expansion into tube sheets and forming into U-bends over small radii.

Substantial quantities of Cufenloy 40 tubes in diameters ranging between $\frac{5}{8}$ and 1 in. and wall thicknesses between 0.049 and 0.120 in. were produced in both the annealed temper (nominal average grain size 0.030 mm) and in the drawn, stress-relieved temper. It was found that tubes so produced and conforming to the minimum properties at room temperature listed in Table I were sufficiently ductile to permit roll expansion and U-bending.

In addition to meeting the standard mercurous nitrate test for stress-corrosion cracking specified in ASTM Specification B 111,[4] specimens of tubes U-bent over a radius equal to twice the diameter of tubes were subjected for 10 months to an even more rigorous test in a moist ammonia, carbon dioxide atmosphere under various conditions of stress on the outer element of the bend. No tendency to stress-corrosion cracking was found in the specimens of this alloy.

It is also worthy of note that the following alloys were tested at the same time and under the same test conditions, with the following results:

90-10 copper-nickel 80-20 copper-nickel 70-30 copper-nickel 65-35 copper-nickel	Did not fail by stress-corrosion cracking during total 10 months' exposure. Specimens were chemically corroded by the test atmosphere. A heavy loose nonprotective scale was formed.
60-40 copper-nickel (Cufenloy 40) 35-65 copper-nickel (monel)	Did not fail by stress-corrosion cracking during total 10 months' exposure. Chemical attack on these specimens was practically nil. A thin, tightly bonded oxide film was formed.

Elevated-temperature tensile and creep properties were determined on specimens made from $\frac{3}{4}$-in. rod stock treated to develop the minimum room-temperature properties listed in Table I. Physical properties such as thermal expansion, thermal conductivity, and electrical resistivity were determined on specimens made from 0.900-in. rod stock of the same material. The composition of the rod stock taken from four different melts, from which these specimens were made, is reported below:

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[4] Specification for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 – 60), 1960 Supplement to Book of ASTM Standards, Part 2, p. 20.

Element	Per Cent
Copper	55.9 to 56.2
Nickel	41.3 to 41.6
Iron	1.92 to 1.96
Manganese	0.45 to 0.58
Phosphorus	0.002 to 0.006
Zinc	0.016 to 0.023

TEST PROCEDURES

Thermal Expansion:

Linear-thermal-expansion measurements were made in a vertical-quartz-tube dilatometer in which dilations were measured by a calibrated dial indicator that had divisions of 0.0001 in. The measurements were made with the specimens in helium at atmospheric pressure. A 3-in.-long cylindrical specimen was used.

Length changes were read from the dial indicator at the same time that temperatures were measured. Each specimen was given two thermal cycles from room temperature to 750 F and back to room temperature. A best curve was drawn through the experimental points, and mean-linear-thermal-expansion coefficients were interpolated over selected temperature ranges from this curve.

The error of the results is estimated not to exceed ±2 per cent.

Thermal Conductivity:

The apparatus and method used in making the thermal-conductivity measurements are essentially the same as in the steady-heat-flow method described by Van Dusen and Shelton (2). The method consists in heating one end of a specimen, measuring the temperature gradients along the specimen, and determining the rate of heat flow through the specimen by means of an ingot iron standard of known thermal conductivity attached to the cold end of the specimen. Radial heat flow into, or away from, the specimen is minimized by thermal insulation and an encircling guard tube in which temperatures are adjusted, as nearly as possible, to match those in the specimen and standard at corresponding levels.

Five 36-gage chromel alumel thermocouples were wedged in holes equally spaced along the specimen, and two similar thermocouples were placed in the ingot iron standard. This permitted the calculation of four thermal-conductivity values, each at a different mean temperature, for each thermal equilibrium. Four thermal equilibria were obtained over a temperature range of approximately 90 to 575 F. A best curve was drawn through these four points, and the thermal conductivity was interpolated at selected temperatures from this curve.

The maximum error of the thermal conductivity values is estimated not to exceed ±5 per cent, the chief uncertainty being the thermal conductivity of the ingot iron standard.

Electrical Resistivity:

Electrical resistivity measurements were made by the voltage-drop method with the specimens protected by a vacuum of approximately 5×10^{-3} mm of mercury. A direct current was passed through the specimen, and voltage drop was measured over a section of the specimen approximately 2 in. long. Measurements were taken with the current flowing normally and reversed, and the values were averaged to minimize thermal and induced potentials.

The error of the electrical resistivity values is estimated not to exceed ±1 per cent.

Tensile Properties:

Tensile properties were determined between 75 and 1050 F by using standard 0.505-in. diameter threaded-end tension specimens. Tests were run in triplicate at each temperature. Specimens were heated in a resistance-wound electric furnace. The atmosphere was static air. Specimen temperature was measured by means of thermocouples and was maintained within ±5 F during each test.

Stress-strain curves were obtained by means of a Baldwin high-magnification extensometer, microformer type, and a standard Baldwin-Southwark Tate-Emery testing machine with an autographic recorder. All specimens were tested at strain rates of 0.003 in. per in. per min to yield and at 0.05 in. per min crosshead speed from yield to fracture.

Modulus of Elasticity:

The modulus of elasticity was determined using the dynamic method. A machined cylindrical specimen $\frac{1}{4}$ in. in diameter and 6 in. long was suspended horizontally from asbestos strings in an electric furnace. The upper end of one string was attached to a loud-speaker diaphragm and the upper end of the other to a crystal pickup. The resonant frequency of the specimen was then used to calculate elastic modulus, as described by Andrew (**3**). Measurements were made between 80 and 1050 F.

Creep and Stress-Rupture Properties:

Creep and stress-rupture properties were determined between 750 and 1050 F by using the same type of specimen as in the tensile-property studies. Tests were conducted in accordance with ASTM Recommended Practice E 139.[5] The specimens were heated in electric-resistance furnaces, in static air, and were held 1 hr at temperature before loading. The desired temperature was maintained within ±2 F. Loading was accomplished in lever-arm creep machines (9:1 lever-arm ratio).

Creep was measured optically. Through windows in the furnace, the movements of two platinum-strip extensometers on opposite sides of the specimen were measured with a filar micrometer microscope. The movements of each extensometer were measured by two observers, and the results of each observer were averaged. With this arrangement, creep movements of less than 0.002 per cent were measured accurately. The zero reading for creep was obtained at temperature before application of the load. Both deformation and time to rupture, if failure occurred at the stress level examined, were measured.

TABLE II.—MEAN LINEAR THERMAL-EXPANSION COEFFICIENTS OF CUFENLOY 40.

Temperature Range, deg Fahr	Mean Linear Thermal Expansion Coefficients per deg Fahr over the Temperature Range Shown
68 to 200	8.2×10^6
68 to 300	8.4
68 to 400	8.6
68 to 500	8.7
68 to 600	8.9
68 to 700	9.0
68 to 750	9.1

Minimum creep rate and total deformation at indicated times were determined from time-deformation curves that were obtained by plotting the individual deformation readings. The creep- and rupture-testing equipment and the procedure used have been described more fully in a previous paper (**4**).

Thermal Stability:

The ability of the alloy to withstand creep exposure without loss of room-temperature properties was measured by determining the tensile properties of unbroken creep specimens after completion of the creep exposure. Test procedures were similar to those employed in determining the tensile properties of the alloy before creep exposure. The specimens were not cleaned (to remove surface oxide) before testing and strength and ductility values were based on the dimensions of the specimen after creep exposure.

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[5] Recommended Practice for Conducting Creep and Time-for-Rupture Tension Tests of Materials (E 139 – 58 T), 1958 Book of ASTM Standards, Part 3, p. 224.

TEST RESULTS

Thermal Expansion:

Linear-thermal-expansion coefficients at selected temperature intervals between 68 and 750 F are given in Table II.

Thermal Conductivity:

Average values for the thermal conductivity are reported in Table III.

TABLE III.—THERMAL CONDUCTIVITY OF CUFENLOY 40.

Temperature, deg Fahr	Thermal Conductivity, Btu per sq ft per hr per deg Fahr per ft
68[a]	12.8
100	13.3
200	14.4
300	15.8
400	17.0
500	18.3
572	19.1

[a] Extrapolated.

Electrical Resistivity:

The electrical resistivity is given in Table IV. These data have also been reported in terms of electrical conductivity. At 68 F, the electrical conductivity of Cufenloy 40 is 3.6 per cent of that of the International Annealed Copper Standard.

Tensile Properties:

The tensile data obtained for Cufenloy 40 finished in the annealed condition are reported in Table V, and those for the drawn and stress-relieved condition in Table VI. The yield strength was measured both at 0.2 per cent offset and 0.5 per cent extension under load. Proportional limit, tensile strength, elongation, and reduction in area were also determined. The tensile strengths and yield strengths (0.5 per cent extension) are plotted as a function of temperature in Fig. 1.

Modulus of Elasticity:

The moduli of elasticity in both conditions are reported in Table VII. The modulus of elasticity of drawn and stress-relieved Cufenloy 40 was greater than that of annealed Cufenloy 40 over the entire temperature range 80 to 1050 F.

TABLE IV.—ELECTRICAL RESISTIVITY AND ELECTRICAL CONDUCTIVITY OF CUFENLOY 40.

Temperature, deg Fahr	Electrical Resistivity, ρ, microhm-cm	$\frac{\rho_T}{\rho_{68}}$[a]	Electrical Conductivity, σ, megmho-cm
68	48.0	1.00	0.0208
100	48.1	1.00	0.0208
200	48.5	1.01	0.0206
300	48.9	1.02	0.0204
400	49.3	1.03	0.0203
500	49.7	1.04	0.0201
572	49.9	1.04	0.0200

[a] $\frac{\rho_T}{\rho_{68}} = \frac{\text{Electrical resistivity at temperature}}{\text{Electrical resistivity at 68 F}}$.

Creep and Stress-Rupture Properties:

Creep and stress-rupture properties in the annealed condition are reported in Table VIII and in the drawn and stress-relieved condition in Table IX. Both minimum creep rate and total deformation as a function of time are included. Time to rupture and ductility at fracture are reported for the stress-rupture tests.

Data contained in Tables VIII and IX have been used to construct the curves shown in Figs. 2 (*a*) and (*b*), which describe the creep and rupture characteristics. The stress-creep rate and stress-rupture time curves were not straight lines on logarithmic coordinates. This type of behavior is typical of copper alloys at elevated temperatures.

TABLE V.—TENSILE PROPERTIES OF CUFENLOY 40 IN THE ANNEALED CONDITION.

Temperature, deg Fahr	Proportional Limit, psi	Yield Strength, psi		Tensile Strength, psi	Elongation in 2 in., per cent	Reduction of Area, per cent
		0.2 per cent Offset	0.5 per cent Extension			
75	...	28 900	29 600	64 900	48	70
75	19 100	28 700	29 700	64 900	49	73
75	19 100	28 900	29 900	64 300	50	72
Average	19 100	28 800	29 700	64 700	49	72
150	17 600	26 900	27 700	60 900	47	73
150	17 500	27 500	28 100	61 900	47	73
150	15 200	27 200	27 900	61 200	47	72
Average	16 800	27 200	27 900	61 300	47	73
200	13 700	26 500	27 500	60 500	46	73
200	16 000	26 300	27 400	60 500	46	72
200	14 500	26 300	27 100	60 200	50	73
Average	14 700	26 400	27 300	60 400	47	73
250	13 000	25 600	26 200	59 100	42	72
250	11 500	25 200	26 700	59 000	45	72
250	11 500	26 000	27 500	59 300	44	73
Average	12 000	25 600	26 800	59 100	44	72
300	11 700	24 900	26 300	58 100	42	72
300	14 700	25 500	26 700	58 000	43	72
300	14 700	27 400	28 700	58 400	40	73
Average	13 700	25 900	27 200	58 200	42	72
350	...	26 400	28 100	56 000	43	72
350	15 300	26 300	28 000	56 800	43	72
350	15 300	26 500	27 800	57 000	42	72
Average	15 300	26 400	28 000	56 600	43	72
400	14 500	25 700	27 000	56 100	41	72
400	15 300	26 500	27 700	56 000	41	70
400	13 700	26 000	27 000	55 900	42	73
Average	14 500	26 100	27 200	56 000	41	72
450	...	25 000	26 700	55 200	41	71
450	12 200	22 400	23 900	55 800	42	72
450	11 400	22 900	24 200	55 600	42	72
Average	11 800	23 400	24 900	55 500	42	72
500	9 900	22 200	23 800	54 400	42	71
500	9 150	22 200	23 800	54 600	41	70
500	9 150	22 600	24 100	54 700	42	70
Average	9 400	22 300	23 900	54 600	42	70
550	11 400	22 400	23 800	54 200	41	67
550	11 400	22 300	23 800	54 100	42	71
550	11 400	22 300	23 700	53 800	40	70
Average	11 400	22 300	23 800	54 000	41	70

TABLE V.—*Continued.*

Temperature, deg Fahr	Proportional Limit, psi	Yield Strength, psi		Tensile Strength, psi	Elongation in 2 in., per cent	Reduction of Area, per cent
		0.2 per cent Offset	0.5 per cent Extension			
600	13 000	22 500	23 900	53 400	40	69
600	11 400	22 500	23 700	53 200	40	68
600	11 400	22 200	23 700	53 200	40	69
Average	11 900	22 400	23 800	53 300	40	69
650	11 400	...	...	53 000	40	66
650	11 400	...	...	52 800	39	67
650	9 900	...	...	53 000	39	65
Average	10 900			52 900	39	66
700	10 000	21 900	23 300	52 300	39	62
700	12 200	22 900	24 400	51 800	38	64
700	12 200	23 000	24 300	51 900	41	65
Average	11 500	22 600	24 000	52 000	39	64
750	18 800	22 500	23 700	51 000	38	59
750	11 400	22 100	23 700	51 000	38	59
750	12 200	22 500	23 800	49 600	37	58
Average	14 100	22 400	23 700	50 500	38	59
800	13 000	22 200	23 700	49 600	34	46
800	13 000	22 100	23 400	49 700	34	50
800	12 200	22 100	23 400	49 800	38	52
Average	12 700	22 100	23 500	49 700	36	49
850	9 900	21 600	22 700	47 100	33	34
850	10 700	21 900	22 900	46 800	32	26
850	11 500	21 900	23 400	46 700	32	28
Average	10 700	21 800	23 000	46 900	32	30
900	9 200	21 600	22 900	43 800	30	32
900	10 700	21 400	22 700	44 600	30	30
900	9 200	21 000	22 400	43 400	27	26
Average	9 700	21 300	22 700	43 900	29	30
950	9 900	21 900	22 900	40 800	27	32
950	9 200	21 900	22 700	40 700	28	27
950	9 900	22 000	23 400	41 600	31	31
Average	9 700	21 900	23 000	41 000	29	30
1000	11 400	20 800	21 900	36 900	29	32
1000	9 200	20 200	21 300	36 800	28	31
1000	10 700	20 600	21 800	36 900	28	30
Average	10 400	20 500	21 700	36 900	28	31
1050	6 900	19 600	20 700	33 800	30	31
1050	9 900	20 000	21 400	34 300	33	35
1050	9 900	20 100	21 200	34 200	36	34
Average	8 900	19 900	21 100	34 100	33	34

TABLE VI.—TENSILE PROPERTIES OF CUFENLOY 40 IN THE DRAWN AND STRESS-RELIEVED CONDITION.

Temperature, deg Fahr	Proportional Limit, psi	Yield Strength, psi		Tensile Strength, psi	Elongation in 2 in., per cent	Reduction of Area, per cent
		0.2 per cent Offset	0.5 per cent Extension			
75	58 500	76 800	76 800	89 700	23	60
75	62 500	76 800	76 800	89 600	22	60
75	60 100	76 000	76 000	88 800	22	60
Average	60 400	76 500	76 500	89 400	22	60
150	48 900	75 300	75 300	86 400	19	59
150	50 900	74 300	74 800	86 200	21	60
150	57 000	73 300	73 800	85 800	22	61
Average	52 300	74 300	74 600	86 100	21	60
200	48 900	72 900	73 000	83 400	19	59
200	...	72 500	72 300	82 400	19	63
200	47 300	72 500	72 500	83 400	21	63
Average	48 100	72 600	72 600	83 100	20	62
250	48 100	71 900	72 000	80 800	20	62
250	51 300	70 200	70 300	80 500	20	62
250	51 700	69 100	69 500	80 600	20	63
Average	50 400	70 400	70 600	80 600	20	62
300	49 900	69 200	69 200	78 600	20	63
300	52 000	70 900	71 000	79 900	20	62
300	50 700	69 300	70 000	78 800	20	62
Average	50 900	69 800	70 100	79 100	20	62
350	45 000	69 600	70 000	78 400	18	61
350	46 400	68 000	67 800	77 300	19	63
350	49 500	67 600	67 600	77 200	20	62
Average	47 000	68 400	68 500	77 600	19	62
400	55 400	69 000	69 000	77 000	18	62
400	46 500	66 600	67 000	76 000	18	61
400	51 900	68 800	68 800	76 500	19	61
Average	51 300	68 100	68 300	76 500	18	61
450	47 700	65 600	65 600	75 000	19	62
450	50 700	66 400	66 500	75 500	19	61
450	49 600	67 800	67 600	75 400	18	60
Average	49 300	66 600	66 600	75 300	19	61
500	50 200	64 800	65 200	73 800	19	62
500	50 800	66 000	66 600	73 800	18	61
500	45 000	66 600	66 700	74 000	18	60
Average	48 700	65 800	66 200	73 900	18	61
550	43 500	65 000	65 400	73 900	18	60
550	43 500	64 800	64 900	73 400	18	59
550	45 700	64 200	64 500	73 500	19	59
Average	44 200	64 700	64 900	73 600	18	59

TABLE VI.—*Continued.*

Temperature, deg Fahr	Proportional Limit, psi	Yield Strength, psi		Tensile Strength, psi	Elongation in 2 in., per cent	Reduction of Area, per cent
		0.2 per cent Offset	0.5 per cent Extension			
600	42 600	65 600	66 000	73 000	18	57
600	42 600	64 500	64 800	73 000	19	59
600	43 500	66 500	66 500	72 800	18	58
Average	42 900	65 500	65 900	72 900	18	58
650	...	...	...	73 700	19	58
650	45 700	65 600	65 600	74 000	17	57
650	48 000	65 500	65 600	73 600	18	58
Average	46 800	65 600	65 600	73 800	18	58
700	45 600	66 400	66 500	73 600	17	56
700	42 700	65 500	65 900	73 600	17	56
700	41 800	65 600	65 600	73 600	18	56
Average	43 700	65 800	66 000	73 600	17	56
750	43 400	64 500	64 700	72 500	17	50
750	38 000	63 900	63 900	71 500	16	49
750	42 700	64 000	64 000	72 000	16	48
Average	41 400	64 100	64 200	72 000	16	49
800	38 200	63 200	63 400	70 600	16	44
800	30 600	62 000	61 200	69 100	16	36
800	30 600	62 400	62 400	69 600	14	38
Average	33 100	62 500	62 300	69 800	15	39
850	37 400	58 800	59 600	66 000	14	31
850	35 900	59 100	58 800	65 400	16	33
850	35 900	58 800	59 100	66 000	13	31
Average	36 400	58 900	59 200	65 800	14	32
900	25 800	54 100	54 100	62 200	15	29
900	29 000	56 000	55 200	62 500	15	32
900	27 500	55 200	54 500	62 400	14	32
Average	27 400	55 100	54 600	62 400	15	31
950	16 000	50 100	51 400	57 500	16	31
950	16 000	50 500	50 800	57 400	17	33
950	15 200	51 000	50 400	57 100	15	33
Average	15 700	50 500	50 900	57 300	16	32
1000	15 200	45 300	46 500	52 300	22	38
1000	13 000	44 800	46 500	52 400	20	36
1000	14 500	45 800	46 900	52 600	20	34
Average	14 200	45 300	46 600	52 400	21	36
1050	9 200	41 800	44 500	48 600	21	36
1050	10 700	40 400	41 700	47 000	16	32
1050	13 000	42 500	42 900	48 400	21	30
Average	11 000	41 600	43 000	48 000	19	33

It was observed that, at stresses where both creep and rupture data were available, the stress-creep rate and the stress-rupture time curves were very nearly mirror images of each other. This observation was applied in extrapolating the stress-rupture time curves from the longest rupture times, about 2000 hr, to determine estimated stress for rupture in 100,000 hr. This method of extrapolation is believed to be much more reliable than a straight-line extrapolation or extrapolation by means of various parameters. The procedure used for extrapolation is in accordance with the work of Monkman and Grant (5), who showed that under certain conditions the product of the creep rate and the rupture time is constant. This relationship was used in the previous paper (1) where it was discussed more fully.

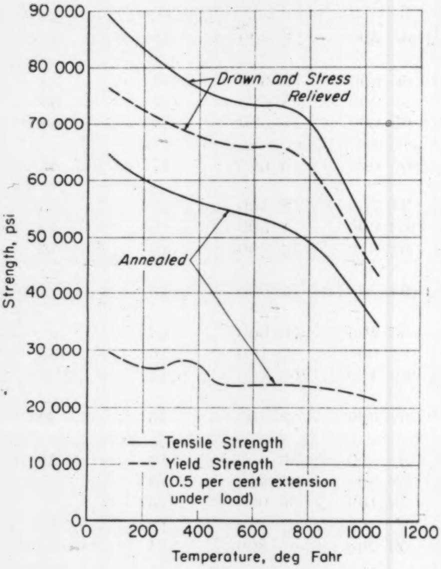

FIG. 1.—Tensile and Yield Strengths of Cufenloy 40 in Two Conditions.

Thermal Stability:

The tensile properties after creep exposure were determined in both conditions and are given in Table X. The material in the annealed condition showed good stability, since all changes observed in tensile properties following creep exposure were directly attributable to plastic straining (work hardening) and not to thermal effects. In the drawn and stress-relieved material, annealing was observed during creep exposure at 1050 F and, to a slight degree, at 950 F. However, the changes in tensile properties at and below 950 F were minor and could be attributed mainly to plastic straining. In no instance was the ductility impaired by thermal exposure.

TABLE VII.—DYNAMIC MODULUS OF ELASTICITY OF CUFENLOY 40.

Temperature, deg Fahr	Modulus of Elasticity, psi	
	Annealed	Drawn and Stress Relieved
80	23.2×10^6	25.3×10^6
150	23.0	25.0
200	22.9	25.0
250	22.7	24.8
300	22.6	24.6
350	22.4	24.4
400	22.1	24.1
450	21.9	24.0
500	21.7	23.6
550	21.5	23.5
600	21.3	23.4
650	21.2	23.2
700	21.0	22.9
750	20.8	22.7
800	20.5	22.5
850	20.3	22.3
900	20.0	22.2
950	19.8	22.1
1000	19.5	21.8
1050	19.3	21.6

DISCUSSION OF RESULTS

The ASME Boiler and Pressure Vessel Code has established criteria for selecting the maximum permissible stress for non-ferrous materials. These criteria, as outlined in Appendix Q of Section VIII of "Rules for Construction of

TABLE VIII.—CREEP AND RUPTURE PROPERTIES OF CUFENLOY 40 IN THE ANNEALED CONDITION.

Temperature, deg Fahr	Stress, psi	Duration, hr	Minimum Creep Rate, per cent per hr	Initial Deformation, per cent	Total Deformation in Indicated Time, per cent						Transition Time,[c] hr	Elongation in 2 in., per cent	Reduction of Area, per cent
					500 hr	1000 hr	1500 hr	2000 hr	2500 hr	3000 hr			
750..........	45 000	23.9[a]	1.4	9.74	...	...	...	...	...	...	...	32.3	32.2
750..........	40 000	187.5[a]	0.080	7.38	...	...	...	...	...	...	...	29.0	27.8
750..........	35 000	3003.0	0.0020	4.06	9.3	10.6	11.6	12.6	13.8	15.4	2400	...	...
750..........	30 000	2714.3	0.00026	2.05	3.84	4.08	4.25	4.38	5.27[b]	...	>2714	...	...
750..........	27 000	2662.6	0.000037	1.63	2.67	2.76	2.80	2.83	2.85	...	>2662	...	...
850..........	40 000	4.7[a]	3.6	8.84	...	...	...	...	...	...	...	38.3	33.3
850..........	33 000	120.1[a]	0.12	3.53	...	...	...	...	...	...	100	22.1	25.6
850..........	30 000	540.6[a]	0.015	2.58	14.3	...	...	...	...	...	430	15.7	20.4
850..........	27 500	1690.4[a]	0.0032	1.36	5.9	7.5	9.9	...	...	...	1050	11.9	15.1
850..........	20 000	2860.3	0.00016	0.170	0.64	0.75	0.85	0.93	1.02	...	>2860	...	...
850..........	12 000	2015.3	0.000012	0.076	0.148	0.157	0.165	0.170	...	...	>2015	...	...
950..........	35 000	2.0[a]	...	5.54	...	...	...	...	...	...	...	37.4	36.0
950..........	25 000	124.8[a]	0.067	1.46	...	...	...	...	...	...	70	17.9	18.8
950..........	22 000	506.3[a]	0.016	0.400	13.1	...	...	...	...	...	270	14.5	11.8
950..........	19 100	1041.6[a]	0.0047	0.192	3.1	8.5	...	...	...	...	450	10.6	10.0
950..........	10 000	1226.5	0.00018	0.076	0.300	0.340	...	...	...	...	>1226	...	...
950..........	3 000	2014.9	0.000019	0.024	0.060	0.070	0.079	0.094	...	...	2000	...	...
1050..........	24 000	6.8[a]	1.6	1.68	...	...	...	...	...	...	3	22.6	22.7
1050..........	18 000	50.1[a]	0.13	0.183	...	...	...	...	...	...	...	12.8	12.9
1050..........	12 000	727.2[a]	0.0065	0.099	4.9	...	...	...	...	...	180	12.8	9.1
1050..........	1 000	1914.5	0.000035	0.004	0.053	0.080	0.098	...	...	...	1600	...	...

[a] Rupture time.

[b] Strain high because of failure of test in adjoining unit.

[c] Beginning of third-stage creep.

TABLE IX.—CREEP AND RUPTURE PROPERTIES OF CUFENLOY 40 IN THE DRAWN AND STRESS-RELIEVED CONDITION.

Temperature, deg Fahr	Stress, psi	Duration, hr	Minimum Creep Rate, per cent per hr	Initial Deformation, per cent	Total Deformation in Indicated Time, per cent						Transition Time,[c] hr	Elongation in 2 in., per cent	Reduction of Area, per cent
					500 hr	1000 hr	1500 hr	2000 hr	2500 hr	3000 hr			
750	65 000	5.6[a]	1.2	0.683	...	...	...	...	...	...	...	11.9	20.9
750	55 000	167.4[a]	0.026	0.397	...	...	...	...	...	...	95	7.2	11.6
750	50 000	544.8[a]	0.007	0.315	4.80	...	...	...	...	...	280	5.5	7.5
750	44 000	3590.0[a]	0.00085	0.264	1.22	1.73	2.15	2.58	3.11	3.76	2150	5.1	5.9
750	40 000	1765.6[a, b]	0.0010	0.243	1.06	1.57	2.18	...	...	...	1950	2.6	3.0
750	35 000	3315.8	0.00009	0.204	0.63	0.74	0.83	0.89	0.94	0.98	>3300	...	...
750	30 000	2519.7	0.000035	0.158	0.326	0.361	0.377	0.395	0.412	...	>2519	...	...
750	25 000	3047.6	0.000012	0.138	0.268	0.296	0.322	0.328	0.334	0.344	>3047	...	...
850	50 000	16.7[a]	0.28	0.372	...	...	...	...	...	...	7	9.4	19.2
850	45 000	77.8[a]	0.057	0.352	...	...	...	...	...	...	34	10.2	11.0
850	39 000	450.6[a]	0.0082	0.284	...	...	...	...	...	...	240	5.5	9.4
850	34 000	1137.7[a]	0.0027	0.251	2.13	4.20	...	...	...	...	650	6.0	5.5
850	31 000	2496.8	0.00034	0.192	0.77	0.94	1.12	1.30	1.53	...	2000	...	...
850	25 000	2473.1	0.00030	0.168	0.71	0.87	1.03	1.18	1.33	...	>2473	...	...
850	15 000	3287.4	0.000035	0.096	0.321	0.355	0.391	0.410	0.428	0.445	>3287	...	...
850	10 000	2346.2	0.000012	0.058	0.130	0.145	0.152	0.158	...	...	>2346	...	...
950	40 000	30.5[a]	0.18	0.349	...	...	...	...	...	...	9	14.0	17.7
950	30 000	401.1[a]	0.013	0.278	...	...	...	...	...	...	150	11.5	10.4
950	25 000	1069.1[a]	0.0048	0.180	2.91	8.30	...	...	...	...	450	10.6	9.4
950	22 000	1504.5[a]	0.0025	0.198	1.82	3.88	...	...	...	...	320	8.9	8.1
950	15 000	1294.2	0.0015	0.118	1.12	1.95	...	...	...	...	850	...	...
950	5 000	1538.8	0.000096	0.028	0.157	0.207	0.255	...	...	...	>1538	...	...
1050	35 000	5.8[a]	1.0	0.411	...	...	...	...	...	...	3	16.6	18.3
1050	25 000	24.7[a]	0.2	0.211	...	...	...	...	...	...	...	9.8	9.2
1050	12 000	520.6[a]	0.013	0.115	13.7	...	...	...	...	...	130	16.2	10.5
1050	8 600	1533.3[a]	0.0047	0.068	2.7	5.9	14.1	...	...	...	350	15.9	10.2
1050	11 000	1538.6	0.00007	0.011	0.115	0.150	0.196	...	...	...	1200	...	...

[a] Rupture time.
[b] Results for this specimen are questionable.
[c] Beginning of third-stage creep.

Unfired Pressure Vessels," 1959, are as follows:

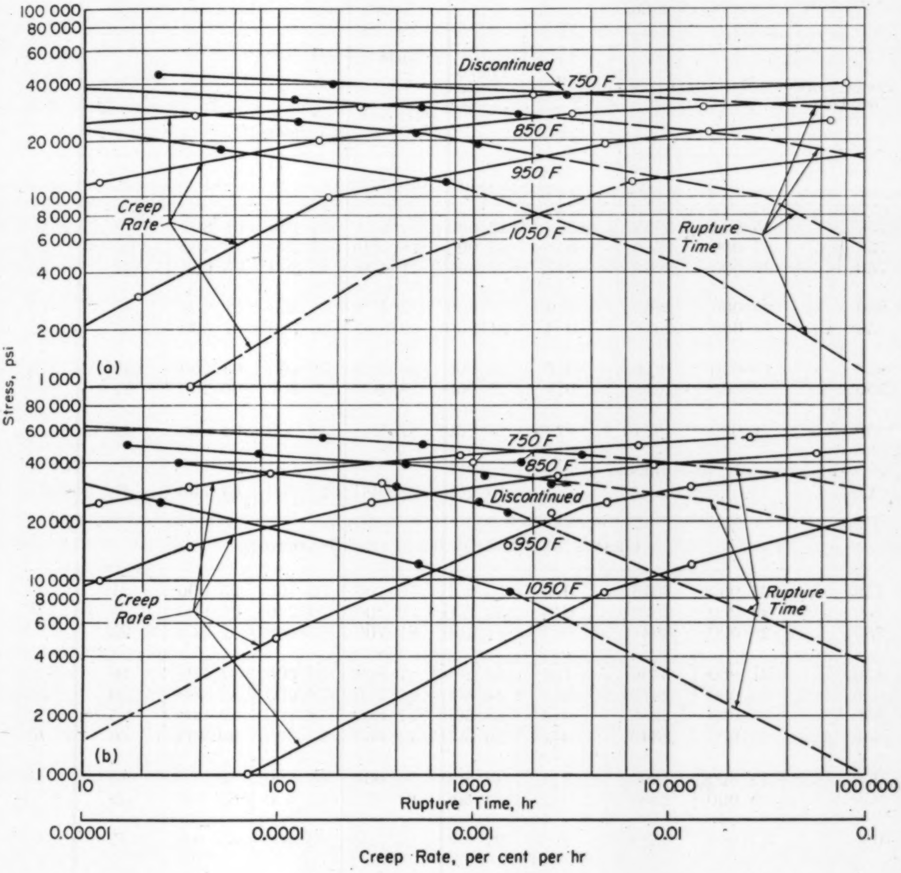

FIG. 2.—Creep and Rupture Characteristics from 750 to 1050 F for Cufenloy 40 (*a*) in the Annealed Condition and (*b*) in the Drawn and Stress-Relieved Condition.

> Except for bolting materials, the maximum allowable stress values may be determined as the lowest of the following when the tensile and yield strengths are obtained from standard short-time tests:
>
> (1) One fourth of the tensile strength, as adjusted to minimum.
> (2) Two thirds of the yield strength (as defined in the material specification) as adjusted to minimum.
> (3) The stress producing a secondary creep rate of 0.1 CRU (creep rupture unit) or 0.1 per cent in 10,000 hr [0.00001 per cent per hr].
> (4) The stress producing rupture in 100,000 hr. (Note—The 1956 Code specified four fifths of the stress for rupture in 100,000 hr.)

The data presented in this paper can be used to select the maximum allowable stress for Cufenloy 40 based on the code requirements.

The tensile strength data in Table V for Cufenloy 40 in the annealed condition were multiplied by 0.989 and the yield-strength data by 0.943 to adjust them to minimum values (the minimum specified tensile and yield strengths at room temperature are 64,000 and 28,000 psi, respectively).

TABLE X.—THERMAL STABILITY OF CUFENLOY 40 IN TWO CONDITIONS.

Creep-Testing Conditions				Tensile Properties After Creep Testing[a]					
Temperature, deg Fahr	Stress, psi	Duration, hr	Plastic Strain, per cent	Proportional Limit, psi	Yield Strength, psi: 0.2 per cent Offset	Yield Strength, psi: 0.5 per cent Extension	Ultimate Strength, psi	Elongation in 2 in., per cent	Reduction of Area, per cent
				ANNEALED CONDITION					
750	35 000	3003	15	42 600	54 800	55 100	61 800	4	19
750	30 000	2714	5	40 000	48 000	48 300	73 400	36	51
750	27 000	2662	2.7	34 400	41 400	42 300	73 300	42	65
850	20 000	2860	0.9	27 600	32 400	33 300	67 000	44	65
850	12 000	2015	0.1	21 900	29 800	30 700	67 500	46	70
950	10 000	1226	0.6	28 000	30 600	31 400	65 500	46	70
950	3 000	2015	0.1	26 900	28 400	29 300	66 200	47	70
1050	1 000	1914	0.1	23 700	27 400	28 200	64 800	48	72
No creep exposure; base tensile properties[b]				19 100	28 800	29 700	64 700	49	72
				DRAWN AND STRESS-RELIEVED CONDITION					
750	35 000	3316	0.8	53 200	76 100	76 100	95 000	21	44
750	30 000	2520	0.3	56 300	79 200	79 200	95 900	25	58
750	25 000	3047	0.2	61 100	81 700	81 400	97 500	25	60
850	31 000	2496	1.4	46 200	67 800	68 600	87 300	19	32
850	25 000	2473	1.2	44 600	69 700	70 000	89 400	20	38
850	15 000	3287	0.4	53 500	73 800	74 100	89 600	25	60
850	10 000	2346	0.1	56 300	71 600	71 600	90 400	26	61
950	15 000	1294	2.5	44 800	64 000	64 500	82 500	16	24
950	5 000	1539	0.2	44 300	61 100	61 600	82 500	29	64
1050	1 000	1539	0.2	22 800	25 400	26 400	65 500	47	72
No creep exposure; base tensile properties[b]				60 400	76 500	76 500	89 400	22	60

[a] All tensile results based on specimen dimensions after creep testing.

[b] Average of three tests.

The tensile strength data in Table VI for Cufenloy 40 in the drawn and stress-relieved condition were multiplied by 0.951 and the yield-strength data by 0.850 to adjust these to minimum values (85,000 and 65,000 psi, respectively). After this was done, the strength properties in the annealed condition were plotted as shown in Fig. 3(*a*), and those for the drawn and stress-relieved condition in Fig. 3(*b*), to establish the maximum allowable stress as outlined by the

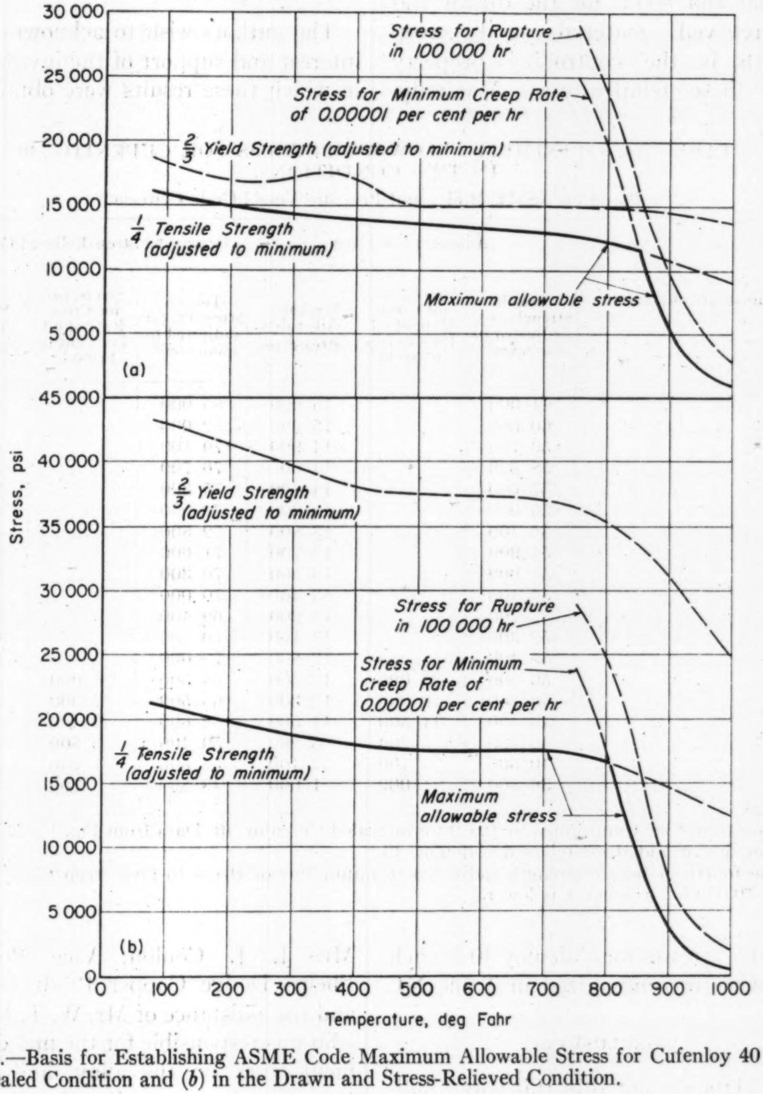

FIG. 3.—Basis for Establishing ASME Code Maximum Allowable Stress for Cufenloy 40 (*a*) in the Annealed Condition and (*b*) in the Drawn and Stress-Relieved Condition.

ASME Code criteria. It is apparent that the tensile strength is the controlling property up to 850 F for the annealed material and 800 F for the drawn and stress-relieved material, and creep strength is the controlling property above these temperatures. Maximum allowable stresses for Cufenloy 40 in each condition are summarized in Table XI.

TABLE XI.—MAXIMUM ALLOWABLE STRESS FOR CUFENLOY 40 IN TWO CONDITIONS.

Based on ASME Boiler and Pressure Vessel Code Criteria.

Temperature, deg Fahr	Annealed Condition			Drawn and Stress-Relieved Condition		
	Tensile Strength, psi (adjusted to minimum)[a]	Stress, psi, for Creep Rate of 0.1 Per Cent in 10,000 hr	Maximum Allowable Stress,[b] psi	Tensile Strength, psi (adjusted to minimum)[a]	Stress, psi, for Creep Rate of 0.1 Per Cent in 10,000 hr	Maximum Allowable Stress,[b] psi
75	64 000	...	16 000	85 000	...	21 200
150	60 600	...	15 200	82 000	...	20 500
200	59 700	...	14 900	79 100	...	19 800
250	58 500	...	14 600	76 700	...	19 200
300	57 600	...	14 400	75 300	...	18 800
350	56 000	...	14 000	73 900	...	18 500
400	55 400	...	13 800	72 800	...	18 200
450	54 900	...	13 700	71 600	...	17 900
500	54 000	...	13 500	70 300	...	17 600
550	53 400	...	13 400	70 000	...	17 500
600	52 700	...	13 200	69 400	...	17 500
650	52 300	...	13 100	70 200	...	17 500
700	51 400	...	12 800	70 000	...	17 500
750	50 000	25 000	12 500	68 500	24 000	17 100
800	49 200	19 200	12 300	66 500	17 300	16 600
850	46 400	11 500	11 500	62 600	9 400	9 400
900	43 400	5 200	5 200	59 400	3 800	3 800
950	40 500	2 100	2 100	54 500	1 500	1 500
1000	36 500	1 000	1 000	49 800	700	700

[a] Data from Fig. 1 multiplied by 0.989 for annealed Cufenloy 40. Data from Fig. 1 multiplied by 0.951 for drawn and stress-relieved Cufenloy 40.

[b] One fourth of tensile strength (adjusted to minimum) or stress to give creep rate of 0.1 per cent in 10,000 hr, whichever is lower.

CONCLUSIONS

The data presented in this paper indicate that Cufenloy 40 in both the annealed and in the drawn and stress-relieved tempers exhibits a suitable combination of strength properties, ductility, and stress-corrosion resistance to make its use advantageous in unfired pressure-vessel applications at temperatures up to 900 F.

Acknowledgments:

The authors wish to acknowledge the interest and support of the investigation in which these results were obtained by Mr. J. J. Conlon, Vice President, Phelps Dodge Copper Products Corp., and the assistance of Mr. W. T. Erickson who was responsible for the mill development work on the alloy, and Mr. L. Caruso under whose direction the stress-corrosion tests were made. On the Battelle staff, Mr. H. W. Deem and Mr. E. H. Eldridge made the physical measurements, Mr. J. E. Campbell the tension tests, and Mr. D. P. Moon the creep and stress-rupture tests.

REFERENCES

(1) W. F. Simmons, B. J. Sirois, D. N. Williams, and R. I. Jaffee, "Properties of 70-30 Copper-Nickel Alloy at Temperatures Ranging Up to 1050 F," *Proceedings*, Am. Soc. Testing Mats., Vol. 59, p. 1035 (1959).

(2) M. S. Van Dusen and S. M. Shelton, "Apparatus for Measuring Thermal Conductivity of Metals Up to 600 C," *Journal of Research*, National Bureau of Standards, Vol. 12, pp. 429–440 (1934).

(3) C. W. Andrews, "Effect of Temperature on the Dynamic Modulus of Elasticity," *Metal Progress*, Vol. 58, pp. 85–89 and 96–100, July, 1950.

(4) W. F. Simmons and J. G. Kura, "The Creep Properties of Three Low-Shrinkage, Copper-Base Casting Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 791 (1958).

(5) F. C. Monkman and N. J. Grant, "An Empirical Relationship Between Rupture Life and Minimum Creep Rate in Creep-Rupture Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 56, p. 593 (1956).

REPORT OF COMMITTEE B-6
ON
DIE-CAST METALS AND ALLOYS*

Committee B-6 on Die-Cast Metals and Alloys held one meeting during the year: in Atlantic City, N. J. on June 29, 1960.

The committee consists of 79 members, of whom 39 are classified as producers, 18 as consumers, and 22 as general interest members.

There is a balance of $3651.84 in the funds of Committee B-6 as of November 30, 1960.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Aluminum-Base Die-Casting Alloys (E. V. Blackmun, chairman) has assigned a task group to collect the available elevated-temperature mechanical property data for aluminum alloy SC84 with various zinc contents. The extent of the data will determine whether it is necessary to undertake such work to obtain needed data for zinc contents at suitable intervals from 1 to 3 per cent at temperatures of 500 to 550 F.

Subcommittee II on Zinc-Base Die-Casting Alloys (A. E. Weiss, chairman) has assigned a task group to determine what procedure to pursue relative to recognizing a new die-casting alloy known in the trade as Zamak-7.

Subcommittee V on Exposure and Corrosion Tests (D. Kleppinger, chairman) has approved reports on Atmospheric Exposure Tests Conducted on Zinc-Alloy Die Castings AC43A, AC41A, and AG40A and Atmospheric Exposure Tests Conducted on Die-Cast Test Bars of Four Magnesium Alloys, as appended hereto.[1] These reports present data and conclusions covering the aforementioned alloys for the 5-, 10-, and 20-yr tests at three outdoor and two indoor locations.

Subcommittee VIII on Brass Die-Casting Alloys (G. F. Hodgson, chairman) is letter balloting on a proposal to increase the allowable maximum iron content from 0.3 to 0.5 per cent in alloy Z30A under Specification B 176 – 57.

This report has been submitted to letter ballot of the committee, which consists of 79 members; 71 members returned their ballots, of whom 64 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. BABINGTON,
Chairman.

GEORGE L. WERLEY,
Secretary.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] See pp. 265 and 273.

APPENDIX I

REPORT OF SUBCOMMITTEE V ON EXPOSURE TESTS OF FOUR DIE-CAST MAGNESIUM ALLOYS

In 1936,[1] Subcommittee VII on Magnesium-Base Die-Casting Alloys recommended that magnesium-base alloys be included in the atmospheric exposure tests proposed for a new series of zinc-base alloys. This program would determine the effect of various exposures on mechanical properties and thereby promote satisfactory application of the metal. In 1937[2] Subcommittee V on Exposure and Corrosion Tests located three producers of magnesium test bars and selected the compositions to be tested as follows:

	CURRENT SIMILAR ALLOY
No. 307 to correspond with then current No. 7 alloy..........	None
No. 312 to correspond with then current No. 12 alloy..........	AM100B
No. 313 (9 per cent Al, 0.6 per cent Zn nominal)..........	AZ91
No. 314 to differ from No. 307 by addition of 2½ per cent zinc...	AZ63

In 1938 Subcommittee V was authorized to include the four above magnesium-base alloys in its exposure program and specified the composition limits as noted in Table I. Casting, metal composition analyses, and original mechanical property determinations were included in the 1939 Report of Committee B-6.[3] The 5- and 10-yr test results listed in subsequent reports[4] are also included in this report.

The exposure period extended from 1939 to 1959.

Summary of Program:

Each producer submitted 225 sets (composed of one die-cast tension test specimen and one die-cast impact bar) of each of four alloy compositions. Of these 5400 total test bars, 3600 were placed on exposure, 240 were used for original property tests and 480 for analyses.

The three producers of the specimens were:

	CODE IDENTIFICATION LETTER
Hoover Co....................	H
Dow Chemical Co..............	K
American Magnesium Corp......	M

Die Casting Technique.—Casting conditions for the test specimens were quite uniform among the producers as shown in Table II.

Chemical Analyses of the alloys were made by The Dow Chemical Co. and The American Magnesium Corp. (Aluminum Company of America) and are listed in Table I with the respective composition specifications.

Exposure Sites.—Bare test bars were placed at ten exposure sites as shown in Table III. Bars were lost from five exposure locations over the 20-yr program amounting to 26½ per cent of the original bars placed on test.

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[1] Report of Committee B-6 on Die-Cast Metals and Alloys, *Proceedings* Am. Soc. Testing Mats., Vol. 36, Part I, p. 178 (1936).

[2] *Ibid.*, Vol. 37, Part I, p. 214 (1937).

[3] *Ibid.*, Vol. 39, p. 277 (1939).

[4] Report of Subcommittee V on Exposure and Corrosion Tests, *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 244 (1946), and Vol. 50, p. 167 (1950).

TABLE I.—CHEMICAL COMPOSITION SPECIFIED[a] AND ACTUAL ANALYSES OF TEST BARS.[b]

Alloy	Producer	Composition, per cent							
		Aluminum	Silicon	Manganese	Zinc	Cadmium	Tin	Copper	Nickel
No. 307....	[a]	6.0 to 7.0	0.5 max	0.15 min	...	...	...	...	...
	H	6.3	0.2	0.14	0	0	0	0.01	0
	K	6.6	0.1	0.24	0	0	0	<0.01	0
	M	6.5	0	0.19	0.1	0	0	<0.01	0
No. 312....	[a]	9.5 to 10.5	1.0 max	0.1 min	...	...	...	...	...
	H	8.9	0.2	0.14	0	0	0	0.01	0
	K	9.4	0.3	0.24	0	0	0	<0.01	0
	M	10.3	0.4	0.14	0	0	0	<0.01	0
No. 313....	[a]	8.5 to 9.5	0.1 max	0.1 min	0.4 to 0.8	...	...	...	...
	H	8.5	0.2	0.14	0.4	0	0	0.01	0
	K	8.6	0	0.22	0.6	0	0	<0.01	0
	M	8.6	0	0.18	0.5	0	0	<0.01	0
No. 314....	[a]	6.0 to 7.0	0.5 max	0.15 min	2.0 to 3.0	...	...	...	...
	H	6.9	0.2	0.14	1.8	0	0	0.02	0
	K	6.4	0	0.33	2.3	0	0	<0.01	0
	M	6.4	0	0.20	2.5	0	0	<0.01	0

[a] Nominal composition specified for these specimens (remainder is magnesium), see *Proceedings*, Am. Soc. Testing Mats., Vol. 38, p. 231 (1938).

[b] Chemical analysis data from *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 280 (1939).

TABLE II.—CASTING CONDITIONS FOR THE DIE-CAST TEST BARS.[a]

Producer	Code	Metal Temperature	Die Temperature	Casting Pressure
The Hoover Co.	H	1180 to 1200 F	250 to 325 F	2180 psi
Dow Chemical Co.	K	1240 to 1290 F	300 to 400 F	2500 psi
American Magnesium Corp.	M	1250 to 1280 F	...	3000 psi

[a] These conditions are different from present-day techniques which generally utilize greater casting pressures of the order of twice or more of the values cited. This could account for the 29,700 psi tensile strength average for alloy No. 313 as compared to the current ASTM Specification B 94 - 58 listing 32,000 to 36,000 psi tensile strength for AZ91A and B alloys.

TABLE III.—TEST SITES AT WHICH THE FOUR MAGNESIUM-BASE ALLOYS WERE EXPOSED IN 1939.

Indoor Atmospheric Exposure	Exposure Period			Environment
	5 yr	10 yr	20 yr	
Coco Solo Islands, C. Z.	×	×	×	Mild tropical
Tucson, Ariz.	×	×	×	Desert
New Kensington, Pa.	×	×	[a]	Laboratory
Cambridge, Mass.	×	[a]	...	Laboratory
Outdoor Atmospheric Exposure				
State College, Pa.	×	×	×	Rural
Key West, Fla.	×	×	[a]	Marine[b]
Sandy Hook, N. J.	×	×	[a]	Marine—industrial
Rochester, N. Y.	[a]	...	...	...
Altoona, Pa.	×	×	×	Industrial
New York, N. Y.	×	×	×	Industrial

[a] Specimens lost before this testing period.

[b] Also see "Key West Corrosion Test Site Reclassified," ASTM BULLETIN, No. 215, July, 1956, p. 58.

TABLE IV.—SUMMARY OF EFFECT OF EXPOSURE ON PROPERTIES OF FOUR MAGNESIUM DIE-CASTING ALLOYS BY EXPOSURE ENVIRONMENTS AND EXPOSURE TIME.

ENVIRONMENT [a]	Number of Exposure Sites	Tensile Strength, psi				Elongation in 2 in., per cent				Hardness, Rockwell E [b]				Charpy Impact Strength, ft-lb				Dimensional Change, in. per in. [c]		
		Original	5 yr	10 yr	20 yr	Orig	5 yr	10 yr	20 yr	Orig	5 yr	10 yr	20 yr	Orig	5 yr	10 yr	20 yr	5 yr	10 yr	20 yr
No. 307 Alloy																				
Indoor............	4	29 800	29 900	28 900	29 300	5	5	4	4	66	65	64	67	3	3	4	3	-0.0001	0.0002	0.0001
Outdoor:																				
Rural............	1	29 800	26 800	25 400	24 400	5	3	4	4	66	63	68	62	3	3	3	2	-0.0001	0.0002	-0.0001
Marine...........	1	29 800	28 800	27 800	d	5	5	4	d	66	66	...	d	3	3	2	d	-0.0001	0.0000	d
Marine-Industrial	1	29 800	26 000	24 100	d	5	3	3	d	66	64	64	d	3	3	2	d	-0.0002	0.0002	d
Industrial.......	2	29 800	24 900	18 500	11 900	5	4	4	3	66	58	59	36	3	3	2[e]	2	-0.0005	0.0022	-0.0001
No. 312 Alloy																				
Indoor............	4	32 000	31 500	31 500	31 500	2	2	2	2	77	86	69	80	1	1	1	1	-0.0001	0.0002	0.0002
Outdoor:																				
Rural............	1	32 000	28 800	30 100	29 300	2	1	2	2	77	78	79	78	1	2	1	1	-0.0001	0.0002	0.0000
Marine...........	1	32 000	29 700	29 700	d	2	1	2	d	77	77	...	d	1	1	1	d	-0.0001	0.0000	d
Marine-Industrial	1	32 000	28 300	28 400	d	2	1	2	d	77	75	76	d	1	1	1	d	-0.0003	0.0002	d
Industrial.......	2	32 000	27 500	21 700	15 400	2	2	2	2	77	75	77	65	1	1	1[e]	1	-0.0005	0.0020	-0.0001
No. 313 Alloy																				
Indoor............	4	29 700	30 200	30 400	31 200	2	2	3	3	73	78	63	76	2	2	2	2	-0.0001	0.0002	0.0002
Outdoor:																				
Rural............	1	29 700	28 700	29 400	27 400	2	2	3	2	73	72	76	73	2	1	1	1	-0.0001	0.0002	-0.0001
Marine...........	1	29 700	30 500	28 600	d	2	3	2	d	73	75	...	d	2	2	1	d	-0.0001	0.0000	d
Marine-Industrial	1	29 700	28 000	26 800	d	2	2	2	d	73	70	74	d	2	2	1	d	-0.0002	0.0002	d
Industrial.......	2	29 700	27 000	20 400	13 000	2	3	3	1	73	69	70	52	2	2	1[e]	2	-0.0004	0.0028	0.0022
No. 314 Alloy																				
Indoor............	4	29 400	32 200	32 300	33 600	4	4	4	3	70	82	66	78	3	3	3	2	-0.0000	0.0002	0.0004
Outdoor:																				
Rural............	1	29 400	30 000	27 100	28 100	4	3	4	4	70	69	74	70	3	2	1	1	-0.0001	0.0003	0.0002
Marine...........	1	29 400	29 500	...	d	4	3	...	d	70	77	...	d	3	2	1	d	-0.0001	0.0001	d
Marine-Industrial	1	29 400	27 700	24 400	d	4	3	3	d	70	67	71	d	3	2	2	d	-0.0002	0.0002	d
Industrial.......	2	29 400	24 900	17 300	10 900	4	3	3	2	70	67	68	31	3	2	1[e]	2	-0.0004	0.0020	0.0014

[a] Indoor Sites: Coco Solo Islands, Canal Zone; New Kensington, Pa.; Cambridge, Mass.; and Tucson, Ariz.
Outdoor Rural Site: State College, Pa.
Outdoor Marine Site: Key West, Fla.
Outdoor Marine-Industrial Site: Sandy Hook, N. J.
Outdoor Industrial Sites: New York, N. Y.; and Altoona, Pa.

[b] Rockwell hardness, E scale (1/8 in. diameter ball, 100-kg load). Hardness conversions were made by use of data from L. B. Grant and A. W. Winston, "Hardness Determinations on Magnesium Alloys," *Metals and Alloys*, May 1930.

[c] *Five Years Exposure.*—The expansion values on the outdoor exposure specimens did not detect whether the bars expanded or contracted upon aging because the specimens were noticeably corroded. Therefore, the values are a measure of the extent of corrosion combined with the aging change.

Ten Years Exposure.—Dimensional measurements have little value for these magnesium alloys because their internal change, if any, was overshadowed by the surface condition of the specimens. Where the surface corrosion products adhered to the specimens, the dimensional change measurements were quite appreciable. Where these corrosion products have corroded away, the measured dimensional changes were of lesser magnitude.

[d] Specimens lost.

[e] Izod impact strength.

[f] V notch 0.050 in. deep having a base radius of 0.010 in. was machined in bars 3 in. in length cut from the center of 6-in. bars.

TABLE V.—EFFECT OF EXPOSURE ON PROPERTIES OF FOUR MAGNESIUM DIE-CASTING ALLOYS.

Average of Three Producers' Properties by Exposure Sites and Alloy Compositions.

Exposure Site [a]	Alloy No.	Tensile Strength, psi				Elongation in 2 in., per cent				Hardness, Rockwell E [b]				Charpy Impact Strength, ft-lb				Dimensional Change, in. per in.[c]		
		Original	5 yr	10 yr	20 yr	Orig	5 yr	10 yr	20 yr	Orig	5 yr	10 yr	20 yr	Orig	5 yr	10 yr	20 yr	5 yr	10 yr	20 yr
Outdoor:																				
New York........	307	29 800	24 600	19 300	10 600	5	4	4	2	66	59	57	28	3	3	2[e]	3	-0.0006	0.0015	0.0015
Altoona.........	307	29 800	25 100	17 400	13 200	5	4	3	4	66	57	61	43	3	3	1[e]	1	-0.0005	0.0029	0.0022
New York........	312	32 000	27 900	22 500	15 300	2	2	2	1	77	72	77	62	1	1	1[e]	1	-0.0006	0.0014	0.0001
Altoona.........	312	32 000	27 000	20 800	15 500	2	2	1	2	77	77	77	68	1	1	1[e]	1	-0.0004	0.0026	0.0012
New York........	313	29 700	27 400	21 800	12 200	2	3	2	1	73	70	71	47	2	2	1[e]	2	-0.0005	0.0016	0.0013
Altoona.........	313	29 700	26 500	18 900	13 800	2	3	3	1	73	68	70	56	2	2	1[e]	1	-0.0003	0.0039	0.0031
New York........	314	29 400	24 000	17 600	9000	4	3	2	1	70	67	69	29	3	2	1[e]	3	-0.0004	0.0016	0.0010
Altoona.........	314	29 400	25 700	17 000	12 700	4	3	3	3	70	67	66	33	3	2	1[e]	1	-0.0003	0.0024	0.0018
Sandy Hook......	307	29 800	26 000	24 100	d	5	3	3	d	66	64	64	d	3	3	2	d	-0.0003	0.0002	d
Key West........	307	29 800	28 800	27 800	d	5	5	4	d	66	66	...	d	3	3	2	d	-0.0001	0.0000	d
State College...	307	29 800	26 800	25 400	24 400	5	3	4	4	66	63	68	62	3	3	3	2	-0.0001	0.0002	-0.0001
Sandy Hook......	312	32 000	28 300	28 400	d	2	1	2	d	77	75	76	d	1	1	1	d	-0.0003	0.0002	d
Key West........	312	32 000	29 700	29 700	d	2	1	2	d	77	77	...	d	1	1	1	d	-0.0001	0.0000	d
State College...	312	32 000	28 800	30 100	29 300	2	1	2	2	77	78	79	78	1	2	1	1	-0.0001	0.0003	-0.0000
Sandy Hook......	313	29 700	28 000	26 800	d	2	2	2	d	73	70	74	d	2	2	1	d	-0.0002	0.0002	d
Key West........	313	29 700	30 500	28 600	d	2	3	2	d	73	75	...	d	2	2	1	d	-0.0001	0.0000	d
State College...	313	29 700	28 700	29 400	27 400	2	2	3	2	73	72	76	73	2	1	1	1	-0.0001	0.0002	-0.0001
Sandy Hook......	314	29 400	27 700	24 400	d	4	3	3	d	70	67	71	d	3	2	2	d	-0.0002	0.0002	d
Key West........	314	29 400	29 500	...	d	4	3	...	d	70	71	...	d	3	2	1	d	-0.0001	0.0001	d
State College...	314	29 400	30 000	27 100	28 100	4	3	4	4	70	69	74	70	3	2	1	1	-0.0001	0.0003	0.0002
Indoor:																				
Coco Solo.......	307	29 800	28 900	27 800	29 200	5	5	5	4	66	67	64	69	3	4	3	3	-0.0000	0.0005	0.0005
New Kensington..	307	29 800	30 300	29 400	d	5	6	4	d	66	62	64	d	3	3	4	d	0.0000	0.0002	d
Cambridge.......	307	29 800	29 900	d	...	5	5	d	...	66	85	d	...	3	0.6[f]	d	...	-0.0000	d	...
Tucson..........	307	29 800	30 400	29 600	29 400	5	5	4	5	66	85	38	66	3	0.4[f]	4	4	-0.0001	0.0000	-0.0001
Coco Solo.......	312	32 000	33 100	31 600	30 600	2	3	3	2	77	80	78	80	1	1	1	1	-0.0001	0.0004	0.0005
New Kensington..	312	32 000	31 300	31 600	d	2	2	2	d	77	76	77	d	1	1	1	d	-0.0001	0.0002	d
Cambridge.......	312	32 000	31 100	d	...	2	1	d	...	77	97	d	...	1	0.3[f]	d	...	-0.0001	d	...
Tucson..........	312	32 000	30 700	32 100	31 500	2	2	2	2	77	91	51	80	1	0.3[f]	1	1	-0.0001	-0.0001	-0.0001
Coco Solo.......	313	29 700	30 300	30 400	31 000	2	3	4	3	73	74	75	77	2	2	2	2	0.0000	0.0005	0.0006
New Kensington..	313	29 700	30 500	30 200	d	2	2	3	d	73	72	73	d	2	2	2	d	-0.0001	0.0002	d
Cambridge.......	313	29 700	30 300	d	...	2	2	d	...	73	81	d	...	2	0.3[f]	d	...	-0.0001	d	...
Tucson..........	313	29 700	29 500	30 500	31 300	2	2	3	3	73	87	40	75	2	0.3[f]	2	2	-0.0001	0.0000	-0.0001
Coco Solo.......	314	29 400	32 700	32 100	33 800	4	5	4	3	70	74	76	80	3	2	3	2	0.0000	0.0005	0.
New Kensington..	314	29 400	32 500	32 500	d	4	5	4	d	70	69	70	d	3	3	3	d	0.0000	0.0002	d
Cambridge.......	314	29 400	32 100	d	...	4	4	d	...	70	92	d	...	3	0.4[f]	d	...	0.0000	d	...
Tucson..........	314	29 400	30 800	32 400	33 300	4	3	3	3	70	86	51	77	3	0.4[f]	2	2	0.0000	0.0000	0.0000

[a] Indoor Sites: Coco Solo Islands, Canal Zone; New Kensington, Pa.; Cambridge, Mass.; and Tucson, Ariz.
Outdoor Rural Site: State College, Pa.
Outdoor Marine Site: Key West, Fla.
Outdoor Marine-Industrial Site: Sandy Hook, N. J.
Outdoor Industrial Sites: New York, N. Y.; and Altoona, Pa.

[b] Rockwell hardness, E scale ($\frac{1}{8}$ in. diameter ball, 100-kg load). Hardness conversions were made by use of data from L. B. Grant and A. W. Winston, "Hardness Determinations on Magnesium Alloys," *Metals and Alloys*, May 1930.

[c] *Five Year Exposure.*—The expansion values on the outdoor exposure specimens did not detect whether the bars expanded or contracted upon aging because the specimens were noticeably corroded. Therefore, the values are a measure of the extent of corrosion combined with the aging change.

Ten Years Exposure.—Dimensional measurements have little value for these magnesium alloys because their internal change, if any, was overshadowed by the surface condition of the specimens. Where the surface corrosion products adhered to the specimens, the dimensional change measurements were quite appreciable. Where these corrosion products have corroded away, the measured dimensional changes were of lesser magnitude.

[d] Specimens lost.

[e] Izod impact strength.

[f] V notch 0.050 in. deep having a base radius of 0.010 in. was machined in bars 3 in. in length cut from the center of 6-in. bars.

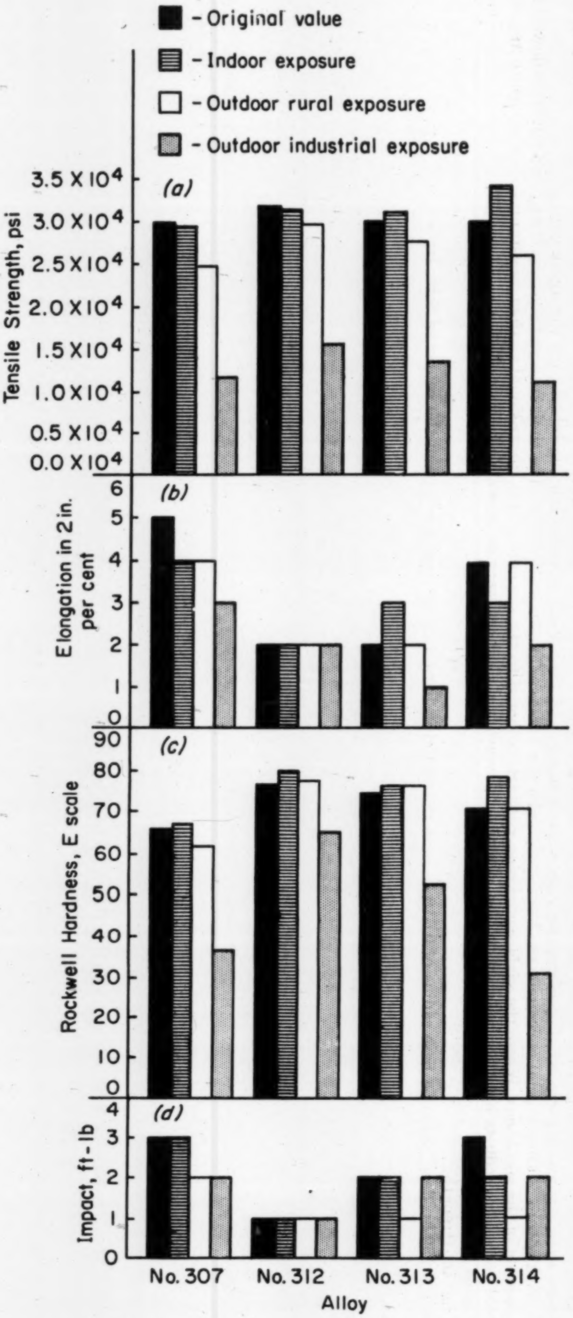

FIG. 1.—Effect of 20 Years Exposure on Tensile Strength, Elongation, Rockwell E Hardness, and Charpy Impact of Magnesium Die Casting Alloys.

Original Properties:

Significant property value differences exist among the various producers which are not readily explainable other than by variations of die casting techniques, except for alloy No. 312 where increasing aluminum contents are logically associated with increasing tensile strengths. Each original property value represents the average of five individual bars.

Properties Versus Exposure:

Values are shown in Tables IV and V[5] for each of the alloys, by producers and for the various exposure periods and exposure sites.

Change in property values was consistent generally within an alloy composition and by producer; that is, original low values would be consistently low for the various exposure periods.

The dimensional change for all four alloys and exposure sites was significantly less than the calculated and laboratory observed value of 0.0008 to 0.0010 in. per in. for maximum total compound precipitation of these compositions of alloys, with the exception of the industrial exposure sites. The relatively larger dimensional increase in measurement associated with the industrial exposure sites is attributed to corrosion product and the inadequacy of the measuring procedure. Therefore, no significant metallurgical dimensional change is believed to have occurred.

Summary of Results:

1. Change in mechanical properties (Fig. 1) was predominantly a function of exposure environment ranging from essentially no change for indoor exposure to considerable reduction for out-door industrial exposure (the most severe condition). (All specimens were tested bare.)

2. All the alloys showed very stable mechanical property values over the 20 years of exposure with the exception of such reductions as were caused by corrosion and consequent decrease in cross-sectional area. (Mechanical property determinations were based on original areas.)

3. The alloys appeared to age slightly proportionate to their zinc contents (alloy No. 313, 0.6 per cent Zn; alloy No. 314, 2½ per cent Zn) as noted by tensile strength and hardness values.

4. Reductions in per cent elongation for the alloys were none or slight with the exception of severe exposure, indicating a uniform type corrosion attack which did not result in pitting or severe notch stress effects.

5. Dimensional stability of the alloys was excellent.

6. Only alloy No. 313 is currently in ASTM Specification B 94 – 58, designated as AZ91 alloy.

Acknowledgments:

In any program of this magnitude, many companies, laboratories, universities and others make very generous contributions in the successful completion of such work. But for such donations it would have been impossible for Subcommittee V to have carried out this important exposure test; therefore, we wish to publicly acknowledge the following participants:

Production of Test Bars

The Hoover Co.
The Dow Chemical Co.
Aluminum Company of America

Analytical Work

The Dow Chemical Co.
Aluminum Company of America

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[5] Detailed results that are summarized in Tables IV and V are not included in the present printing; copies may be secured on request from ASTM Headquarters (Research Report file No. RR 9: B-6).

Mechanical Testing

Aluminum Company of America
The Dow Chemical Co.
The New Jersey Zinc Co.
Battelle Memorial Inst.
General Electric Co.
Westinghouse Air Brake Co.
Sperry Gyroscope Co.
Chrysler Corp.
Stewart Die Casting Division, Stewart-Warner Corp.
The Hoover Co.
Hampden Brass and Aluminum Co.
Bell Telephone Laboratories Inc.
Apex Smelting Co.

Exposure Services

Bell Telephone Laboratories Inc.
Pennyslvania Railroad
Pennsylvania State University
University of Arizona
L. B. Sartain

This report was prepared for the subcommittee by F. L. Burkett, Donald Kleppinger, and G. L. Werley.

Respectfully submitted on behalf of the subcommittee,

DONALD KLEPPINGER,
Chairman.

APPENDIX II

REPORT OF SUBCOMMITTEE V ON ATMOSPHERIC EXPOSURE TESTS OF ZINC-ALLOY DIE CASTINGS AC43A, AC41A, AND AG40A

During the meeting of Subcommittee V on Exposure and Corrosion Tests held on March 5, 1936, it was decided that a second series of exposure tests on zinc-alloy die castings should be undertaken, since the test results from the first exposure test had little remaining value in that all of the alloys in that program had become obsolete in the interim. There was then a need for similar atmospheric exposure data on the three new commercial zinc die-casting alloys AC43A, AC41A, and AG40A[1] (Tables I and II).

Alloy AC41A was a relatively new alloy at that time and it was not until June 1936 that Subcommittee II recommended its adoption and inclusion in ASTM Specification B 86.[1] The following explanatory notes regarding the differences for the three new alloys were appended to the Subcommittee II minutes:

> Alloy AC43A has the highest tensile strength and hardness. It has better corrosion resistance than alloy AG40A but undergoes a slow loss of impact strength and an increase in dimensions when aged at room temperature. These changes are accelerated to a marked extent by an increase in service temperature.
>
> Alloy AC41A is intermediate between AC43A and AG40A in tensile strength and hardness. Its corrosion resistance is equal to that of alloy AC43A. At room temperature, it undergoes little, if any, change in impact strength and dimensions. At elevated temperatures, a slow loss in impact strength and a small growth in dimensions occurs.
>
> Alloy AG40A undergoes little, if any, change in impact strength or dimensions during aging at room or elevated temperature. This alloy is, however, somewhat softer and lower in tensile strength than alloy AC43A and is somewhat more susceptible to corrosion.

Upon completion of the 20-yr exposure tests, the average mechanical properties of the three alloys confirmed the predicted differences, as can be seen from the data in Table I.

Location and Duration of Atmospheric Tests:

Atmospheric tests of 5-, 10-, and 20-yr duration were planned for the following exposure locations:

Outdoors

State College, Pa.
Key West, Fla.[a]
Sandy Hook, N. J.[a]
Rochester, N. Y.[b]
Altoona, Pa.
New York, N. Y.

Indoors

Coco Solo Islands, C. Z.
Tucson, Ariz.
New Kensington, Pa.[a]
Cambridge, Mass.[c]

---

[1] Alloys AC41A and AG40A (when this work was initiated, these alloys were designated XXIII and XXV, respectively) appear in Specifications for Zinc-Base Alloy Die Castings (B 86 – 57 T). Alloy AC43A (formerly alloy XXI) appeared in Specifications B 86 – 53 T.

[a] At the end of 20 yr, further losses had occurred at Key West, Sandy Hook, and New Kensington. Therefore, the planned atmospheric tests were only completed at 3 outdoor exposures (State College, Altoona, and New York); and 2 indoor exposures (Coco Solo and Tucson).

[b] All specimens were lost before the end of the first exposure period at Rochester, N. Y.

[c] At the end of 10 years, the specimens at Cambridge, Mass., had also been lost.

TABLE I.—AVERAGE CHANGES IN PROPERTIES FOR ALLOYS AC43A, AC41A, AND AG40A DURING 20 YEARS' EXPOSURE AT THREE OUTDOOR AND TWO INDOOR LOCATIONS.

Alloys	Original	After 20 Years	Change Per Cent
TENSILE STRENGTH, PSI			
AC43A	48 600	43 000	−12
AC41A	44 300	34 500	−22
AG40A	36 900	31 700	−14
IMPACT STRENGTH, FT-LB			
AC43A	29	5	−83
AC41A	41	18	−56
AG40A	39	28	−28
ROCKWELL E HARDNESS			
AC43A	96	90	−6
AC41A	91	80	−12
AG40A	83	72	−13
ELONGATION IN 2 IN., PER CENT			
AC43A	10	2	−80
AC41A	7	8	+14
AG40A	15	14	−7
DIMENSIONAL CHANGE, IN. PER IN.			
AC43A	...	+0.0014	+0.14
AC41A	...	−0.0001	−0.01
AG40A	...	−0.0001	−0.01

TABLE II.—ALLOY COMPOSITIONAL LIMITS.

	AC43A (XXI)	AC41A (XXV)	AG40A (XXIII)[a]
Aluminum, per cent	3.5 to 4.5	3.5 to 4.3	3.5 to 4.3
Copper, per cent	2.5 to 3.5	0.75 to 1.25	0.03 max
Magnesium, per cent	0.02 to 0.10	0.03 to 0.08	0.03 to 0.08
Lead, per cent	0.007 max	0.007 max	0.007 max
Cadmium, per cent	0.005 max	0.005 max	0.005 max
Tin, per cent	0.005 max	0.005 max	0.005 max
Iron, per cent	0.100 max	0.100 max	0.100 max
Zinc, per cent	remainder	remainder	remainder

---

[a] Specimens of alloy AG40A from two producers were not included in any of the tests of this program because they were off grade in chemical composition.

Producers of Test Specimens:	*Code Identification Letter*
Apex Smelting Co.	A
Doehler-Jarvis Division, National Lead Co.	D
The Bunker Hill Co.	J
The Superior Die Casting Co.	P
Stewart Die Casting Div., Stewart-Warner Corp.	S
The New Jersey Zinc. Co.	Z

Die Casting Technique:

All test bars were die cast in standard dies[2] and under the following standard casting conditions:

Calculated pressure on metal	In excess of 1000 psi
Metal Temperature	Not over 800 F
Die Temperature	Above 350 F

Original Properties:

All original mechanical property tests were scheduled to be made after a minimum of three months' aging. This practice was agreed upon since it was known to be impossible for all producers to prepare their test bars at the same time but still a sound comparison basis was necessary. Actually, the die casting was done over a period of a month; therefore, no bars were aged four months while adhering to the minimum of three months. This minor aging spread had no significant effect on the original properties.

There were significant differences, however, in the properties of the bars for the various producers, some of which are explainable on the basis of allowable variations in composition. For instance, the spread in tensile strength for the different producers of alloy AC43A is due to the variations in their copper contents. The highest tensile strength appeared in the *S* bars which had the highest copper content (3.0 per cent), and the lowest tensile strength occurred in the *P* bars which had the lowest copper content (2.6 per cent). All other producers had intermediate copper contents and intermediate tensile strengths.

Properties After 20-yr Aging:

AC43A exhibited the predicted embrittlement and dimensional growth, either of which are undesirable in many die casting applications. These factors were responsible for the obsolescence of the alloy and its removal from the ASTM Specification B 86 prior to the completion of the 20-yr exposure tests.

AC41A and AG40A both exhibited a high degree of permanence of mechanical properties and an almost complete stability of dimensions during 20 yr of severe atmospheric conditions, including seacoast and tropical exposures.

The individual test results including those for the intermediate exposure periods of 5 and 10 yr are tabulated in Tables III, IV, and V.

Acknowledgments:

In any program of this magnitude many companies, laboratories, universities and their kind make very generous contributions in the successful completion of such work. But for such donations it would have been impossible for Subcommittee V to have carried out this important exposure test; therefore, we wish to publicly acknowledge the following participants:

Production of Test Bars

Apex Smelting Co.
Doehler-Jarvis Div., National Lead Co.
The Bunker Hill Co.
The Superior Die Casting Co.
Stewart Die Casting Div., Stewart-Warner Corp.
The New Jersey Zinc Co.

Analytical Work

Bell Telephone Laboratories, Inc.
Chrysler Motors Corp.
The New Jersey Zinc Co.

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[2] G. L. Werley, "A Study of Die Design Changes for the Improvement of the Soundness and Uniformity of Test Bars," *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part 1, p. 223 (1937).

TABLE III.—EFFCT OF EXPOSURE UPON PROPERTIES OF DIE-CAST AC43A ALLOY.

Producer	Tensile Strength, psi				Elongation in 2 in., per cent				Hardness, Rockwell E				Charpy Impact Strength, ft-lb				Dimensional Change, in. per in.		
	Orig.	5 yr	10 yr	20 yr	Orig.	5 yr	10 yr	20 yr	Orig.	5 yr	10 yr	20 yr	Orig.	5 yr	10 yr	20 yr	5 yr	10 yr	20 yr
Exposed Outdoors at New York, N. Y.																			
A	47 700	47 300	44 000	37 400	11	6	3	1	95	93	97	76	26	15	9	8	0.0002	0.0007	0.0011
D	49 800	47 800	44 000	35 100	5	3	1	1	98	96	100	75	31	12	3	8	0.0003	0.0009	0.0011
J	47 000	47 000	43 200	37 100	8	5	3	1	94	95	98	75	30	19	6	5	−0.0001	0.0007	0.0008
P	45 700	43 800	39 800	36 200	7	8	4	2	95	93	97	80	26	23	15	5	−0.0001	0.0004	0.0007
S	52 300	55 200	45 200	40 200	16	3	3	1	97	99	101	73	23	4	1	10	0.0006	0.0014	0.0022
Z	49 100	49 700	46 100	41 100	12	5	3	2	96	98	101	74	38	15	5	7	0.0003	0.0010	0.0017
Average	48 600	48 500	43 700	37 800	10	5	3	1	96	96	99	76	29	15	6	7	0.0002	0.0008	0.0013
Exposed Outdoors at Altoona, Pa.																			
A	47 700	46 300	41 900	37 000	11	7	1	1	95	95	81	94	26	16	9	1	0.0002	0.0008	0.0011
D	49 800	47 800	42 300	34 300	5	4	1	1	98	98	84	94	31	17	5	1	0.0003	0.0010	0.0011
J	47 000	45 600	41 700	36 900	8	6	1	1	94	95	80	94	30	17	8	1	0.0000	0.0003	0.0005
P	45 700	42 100	39 100	35 900	7	8	2	1	95	95	82	93	26	20	18	3	0.0000	0.0005	0.0003
S	52 300	55 200	48 700	39 000	16	6	1	0	97	100	88	96	23	4	2	1	0.0004	0.0012	0.0019
Z	49 100	48 900	45 400	39 800	12	9	2	1	96	98	84	96	38	21	6	1	0.0002	0.0008	0.0014
Average	48 600	47 600	43 200	37 200	10	7	1	1	96	97	83	94	29	16	8	1	0.0002	0.0008	0.0010
Exposed Outdoors at State College, Pa.																			
A	47 700	47 100	46 500	47 800	11	5	3	2	95	99	92	92	26	15	6	10	0.0001	0.0004	0.0009
D	49 800	49 100	47 800	46 400	5	3	1	1	98	100	93	94	31	9	2	2	0.0002	0.0006	0.0010
J	47 000	47 200	47 200	48 800	8	5	3	2	94	99	83	94	30	17	6	7	−0.0001	0.0001	0.0005
P	45 700	43 900	42 900	45 300	7	6	4	4	95	97	91	90	26	20	11	18	−0.0002	0.0001	0.0004
S	52 300	57 200	53 600	50 500	16	3	2	2	97	102	94	95	23	5	2	6	0.0004	0.0009	0.0017
Z	49 100	50 300	50 000	51 400	12	6	4	3	96	101	95	95	38	17	7	6	0.0001	0.0005	0.0012
Average	48 600	49 100	48 000	48 400	10	5	3	2	96	100	91	93	29	14	6	8	0.0001	0.0004	0.0010

Exposed Indoors at Coco Solo Islands, C. Z.

A	47 700	49 600	46 200	44 400	11	7	4	3	95	89	95	90	26	4	3	1	0.0010	0.0017	0.0023
D	49 800	48 400	44 900	43 800	5	2	3	2	98	90	93	92	31	4	3	1	0.0012	0.0018	0.0020
J	47 000	49 900	47 200	45 000	8	4	3	2	94	90	93	87	30	5	3	1	0.0006	0.0012	0.0019
P	45 700	47 100	42 700	43 600	7	5	3	2	95	89	94	89	26	7	4	1	0.0006	0.0011	0.0019
S	52 300	53 400	49 000	47 800	16	3	4	3	97	91	96	92	23	2	3	1	0.0017	0.0024	0.0026
Z	49 100	53 600	49 800	46 100	12	5	4	2	96	92	96	90	38	4	3	1	0.0012	0.0019	0.0024
Average	48 600	50 300	46 600	45 100	10	4	4	2	96	90	94	90	29	4	3	1	0.0011	0.0017	0.0022

Exposed Indoors at Tucson, Ariz.

A	47 700	51 100	48 300	46 500	11	4	3	4	95	95	97	95	26	6	3	3	0.0008	0.0016	0.0020
D	49 800	50 700	48 500	46 400	5	3	2	2	98	97	98	97	31	6	4	6	0.0010	0.0014	0.0017
J	47 000	51 700	49 500	46 800	8	3	2	3	94	96	97	96	30	9	4	3	0.0004	0.0009	0.0015
P	45 700	46 000	47 000	44 100	7	4	3	3	95	94	97	95	26	13	7	8	0.0002	0.0008	0.0012
S	52 300	55 600	52 600	48 600	16	4	5	6	97	97	97	95	23	2	2	4	0.0015	0.0021	0.0023
Z	49 100	55 000	51 700	47 800	12	6	3	2	96	98	98	96	38	13	6	6	0.0009	0.0017	0.0020
Average	48 600	51 700	49 600	46 700	10	4	3	3	96	96	97	96	29	8	4	5	0.0008	0.0014	0.0018

Average of Above Five Exposures for Each Producer

A	47 700	48 300	45 400	42 600	11	6	3	2	95	94	92	89	26	11	6	5	0.0005	0.0010	0.0015
D	49 800	48 800	45 500	41 200	5	3	2	1	98	96	94	90	31	10	3	4	0.0006	0.0011	0.0014
J	47 000	48 300	45 800	42 900	8	5	2	2	94	95	90	89	30	13	5	3	0.0002	0.0006	0.0010
P	45 700	44 600	42 300	41 000	7	6	3	2	95	94	92	89	26	17	11	7	0.0001	0.0006	0.0009
S	52 300	55 300	49 800	45 200	16	4	3	2	97	98	95	90	23	3	2	4	0.0009	0.0016	0.0021
Z	49 100	51 500	48 600	45 200	12	6	3	2	96	97	95	90	38	14	5	4	0.0005	0.0010	0.0017
Grand Average	48 600	49 500	46 200	43 000	10	5	3	2	96	96	93	90	29	11	6	5	0.0005	0.0010	0.0014

TABLE IV.—EFFECT OF EXPOSURE UPON PROPERTIES OF DIE-CAST AC41A ALLOY.

Producer	Tensile Strength, psi				Elongation in 2 in., per cent				Hardness, Rockwell E				Charpy Impact Strength, ft-lb				Dimensional Change, in. per in.		
	Orig.	5 yr	10 yr	20 yr	Orig.	5 yr	10 yr	20 yr	Orig.	5 yr	10 yr	20 yr	Orig.	5 yr	10 yr	20 yr	5 yr	10 yr	20 yr
EXPOSED OUTDOORS AT NEW YORK, N. Y.																			
A	44 100	40 100	36 000	30 600	10	7	5	4	92	86	89	76	42	39	27	12	−0.0002	0.0000	−0.0001
D	46 700	39 300	36 800	31 700	4	3	3	5	92	87	90	70	44	42	26	4	−0.0001	0.0002	−0.0002
J	42 200	37 800	34 100	29 300	6	6	4	3	89	83	87	73	41	38	19	5	−0.0002	0.0000	−0.0002
P	42 900	38 100	34 800	30 500	6	9	5	5	89	82	87	69	38	35	28	14	−0.0003	0.0000	−0.0003
S	45 600	41 400	36 500	31 600	8	8	5	4	91	85	89	74	37	34	22	3	−0.0001	0.0002	−0.0001
Z	44 200	39 800	35 500	30 300	9	10	6	4	91	85	89	72	46	41	29	9	−0.0002	0.0003	0.0000
Average	44 300	39 400	35 600	30 700	7	7	5	4	91	85	88	72	41	38	25	8	−0.0002	0.0001	−0.0002
EXPOSED OUTDOORS AT ALTOONA, PA.																			
A	44 100	37 300	34 000	30 300	10	6	3	2	92	88	75	83	42	29	26	13	−0.0002	0.0000	−0.0001
D	46 700	39 100	35 500	30 700	4	5	2	3	92	90	77	84	44	30	23	13	−0.0001	0.0000	−0.0002
J	42 200	35 700	32 600	28 600	6	6	3	2	89	86	72	81	41	21	19	3	−0.0002	−0.0001	−0.0003
P	42 900	36 200	33 700	29 900	6	9	4	3	89	86	73	83	38	28	24	16	−0.0003	0.0000	−0.0004
S	45 600	39 600	36 200	32 100	8	9	3	3	91	89	74	84	37	24	18	4	−0.0002	0.0002	0.0000
Z	44 200	37 300	34 600	30 800	9	8	4	4	91	89	74	85	46	33	24	10	−0.0002	0.0002	0.0000
Average	44 300	37 500	34 400	30 400	7	7	3	3	91	88	74	83	41	28	22	10	−0.0002	0.0000	−0.0002
EXPOSED OUTDOORS AT STATE COLLEGE, PA.																			
A	44 100	40 100	39 000	37 800	10	8	5	8	92	90	83	81	42	35	26	33	−0.0002	−0.0001	−0.0002
D	46 700	42 200	39 800	38 300	4	5	3	6	92	93	86	82	44	37	16	28	−0.0001	−0.0001	−0.0001
J	42 200	38 500	37 400	36 300	6	7	5	6	89	90	81	78	41	33	30	37	−0.0002	−0.0002	−0.0002
P	42 900	39 100	38 100	36 800	6	8	6	10	89	90	83	78	38	33	30	40	−0.0003	−0.0003	−0.0004
S	45 600	42 300	40 300	39 000	8	10	6	10	91	92	81	80	37	32	28	30	−0.0001	−0.0001	−0.0001
Z	44 200	40 200	38 700	37 400	9	9	8	14	91	93	84	79	46	41	18	31	−0.0002	−0.0002	−0.0001
Average	44 300	40 400	38 900	37 600	7	8	6	9	91	91	83	80	41	35	25	33	−0.0002	−0.0002	−0.0002

EXPOSED INDOORS AT COCO SOLO ISLANDS, C. Z.

A	44 100	38 700	36 600	36 600	10	20	17	11	92	78	84	80	42	35	35	16	−0.0001	−0.0001	0.0000
D	46 700	40 100	37 400	35 300	4	10	10	9	92	85	84	82	44	37	35	12	−0.0001	0.0000	0.0001
J	42 200	36 900	35 900	36 100	6	9	9	6	89	80	82	77	41	31	32	15	−0.0002	−0.0001	0.0001
P	42 900	38 300	36 300	35 800	6	13	17	14	89	82	84	79	38	30	36	25	−0.0003	−0.0003	−0.0001
S	45 600	39 800	38 100	37 900	8	11	17	7	91	82	86	81	37	29	28	4	−0.0001	0.0000	0.0000
Z	44 200	38 200	37 000	36 600	9	21	15	9	91	82	85	81	46	40	36	10	−0.0001	0.0000	0.0002
Average	44 300	38 700	36 900	36 400	7	14	14	9	91	82	84	80	41	34	34	14	−0.0002	−0.0001	0.0000

EXPOSED INDOORS AT TUCSON, ARIZ.

A	44 100	41 000	38 100	37 600	10	12	13	19	92	86	88	85	42	38	31	20	−0.0002	−0.0001	−0.0001
D	46 700	39 900	39 600	36 400	4	7	14	3	92	87	88	88	44	41	38	31	−0.0001	−0.0001	−0.0001
J	42 200	39 200	37 600	36 600	6	6	8	9	89	83	86	85	41	35	34	25	−0.0002	−0.0002	−0.0001
P	42 900	39 800	38 000	36 800	6	15	12	20	89	84	87	85	38	41	38	35	−0.0004	−0.0003	−0.0004
S	45 600	41 900	39 300	38 900	8	16	8	20	91	83	87	85	37	32	29	13	−0.0001	−0.0001	−0.0001
Z	44 200	39 900	38 700	37 600	9	17	12	19	91	86	87	86	46	44	38	35	−0.0002	−0.0001	−0.0001
Average	44 300	40 300	38 600	37 300	7	12	11	15	91	85	87	86	41	38	35	26	−0.0002	−0.0002	−0.0002

AVERAGE OF ABOVE FIVE EXPOSURES FOR EACH PRODUCER

A	44 100	39 400	36 700	34 600	10	11	9	9	92	86	84	81	42	35	29	19	−0.0002	−0.0001	−0.0001
D	46 700	40 100	37 800	34 500	4	6	6	5	92	88	85	81	44	37	28	18	−0.0001	0.0000	−0.0001
J	42 200	37 600	35 500	33 400	6	7	6	5	89	84	82	79	41	32	27	17	−0.0002	−0.0001	−0.0001
P	42 900	38 300	36 200	34 000	6	11	9	10	89	85	83	79	38	33	31	26	−0.0003	−0.0002	−0.0003
S	45 600	41 000	38 100	35 900	8	11	8	9	91	86	83	81	37	30	25	11	−0.0001	0.0000	−0.0001
Z	44 200	39 100	36 900	34 500	9	13	9	10	91	87	84	81	46	40	29	19	−0.0002	0.0000	0.0000
Grand Average	44 300	39 300	36 900	34 500	7	10	8	8	91	86	83	80	41	35	28	18	−0.0002	0.0000	−0.0001

TABLE V.—EFFECT OF EXPOSURE UPON PROPERTIES OF DIE-CAST AG40A ALLOY.

Producer	Tensile Strength, psi				Elongation in 2 in., per cent				Hardness, Rockwell E				Charpy Impact Strength, ft-lb				Dimensional Change, in. per in.		
	Orig.	5 yr	10 yr	20 yr	Orig.	5 yr	10 yr	20 yr	Orig.	5 yr	10 yr	20 yr	Orig.	5 yr	10 yr	20 yr	5 yr	10 yr	20 yr
Exposed Outdoors at New York, N. Y.																			
A	37 000	34 100	31 500	29 400	17	7	9	4	84	77	81	70	33	38	32	8	−0.0001	−0.0001	−0.0003
J	36 500	34 100	30 800	28 700	12	7	11	4	82	77	81	63	40	41	31	6	−0.0001	−0.0002	−0.0003
P	36 500	34 100	30 400	29 000	15	13	24	8	82	77	80	66	41	44	33	7	−0.0002	−0.0002	−0.0002
Z	37 700	35 000	31 600	29 400	15	10	18	6	85	79	82	71	43	44	30	17	−0.0001	−0.0001	−0.0002
Average	36 900	34 300	31 100	29 100	15	9	16	6	83	78	81	68	39	42	32	10	−0.0001	−0.0002	−0.0002
Exposed Outdoors at Altoona, Pa.																			
A	37 000	33 600	32 000	30 600	17	19	6	5	84	79	68	73	33	29	43	12	−0.0001	−0.0001	−0.0002
J	36 500	33 100	32 100	30 000	12	12	6	4	82	76	71	75	40	26	44	11	−0.0001	−0.0002	−0.0003
P	36 500	32 800	32 200	30 800	15	24	11	13	82	76	70	75	41	28	43	15	−0.0001	−0.0002	−0.0002
Z	37 700	34 000	33 700	30 900	15	18	12	9	85	80	73	77	43	36	46	20	−0.0002	0.0000	−0.0001
Average	36 900	33 400	32 500	30 600	15	18	9	8	83	78	70	75	39	30	44	14	−0.0001	−0.0001	−0.0002
Exposed Outdoors at State College, Pa.																			
A	37 000	35 500	33 600	32 800	17	15	9	18	84	82	75	67	33	39	28	33	−0.0001	0.0000	0.0000
J	36 500	35 000	33 400	32 500	12	12	7	12	82	82	75	67	40	37	33	39	−0.0001	0.0000	−0.0001
P	36 500	34 200	33 400	32 100	15	21	15	26	82	81	74	65	41	33	33	38	−0.0001	0.0000	0.0000
Z	37 700	35 600	33 900	33 200	15	18	13	16	85	85	75	68	43	41	21	42	0.0000	0.0000	0.0000
Average	36 900	35 100	33 600	32 600	15	16	11	18	83	82	75	67	39	38	29	38	−0.0001	0.0000	0.0000
Exposed Indoors at Coco Solo Islands, C. Z.																			
A	37 000	33 800	32 500	32 500	17	19	22	22	84	68	77	72	33	26	42	39	0.0000	0.0000	0.0000
J	36 500	33 600	32 500	32 200	12	16	22	21	82	68	75	70	40	38	43	34	0.0000	0.0000	0.0000
P	36 500	34 100	31 900	31 700	15	25	28	25	82	68	76	70	41	39	44	35	0.0000	0.0000	0.0002
Z	37 700	33 800	33 100	33 500	15	19	20	16	85	68	78	72	43	26	44	40	0.0000	0.0000	0.0000
Average	36 900	33 800	32 500	32 500	15	20	23	21	83	68	76	71	39	32	43	37	0.0000	0.0000	0.0000
Exposed Indoors at Tucson, Ariz.																			
A	37 000	36 100	34 200	33 400	17	16	20	20	84	76	79	77	33	43	38	38	−0.0001	−0.0001	−0.0001
J	36 500	36 000	33 800	33 600	12	12	14	17	82	77	79	77	40	41	40	42	−0.0001	−0.0001	−0.0002
P	36 500	35 500	33 700	33 100	15	21	23	24	82	77	79	77	41	40	40	44	−0.0001	−0.0001	−0.0001
Z	37 700	36 500	34 800	34 200	15	15	17	17	85	77	80	78	43	47	40	43	−0.0001	−0.0001	−0.0001
Average	36 900	36 000	34 100	33 600	15	16	18	20	83	77	79	77	39	43	40	42	−0.0001	−0.0001	−0.0001
Average of Above Five Exposures for Each Producer																			
A	37 000	34 600	32 800	31 700	17	15	13	14	84	76	76	72	33	35	37	26	−0.0001	−0.0001	−0.0001
J	36 500	34 400	32 500	31 400	12	12	12	12	82	76	76	70	40	37	38	26	−0.0001	−0.0001	−0.0002
P	36 500	34 100	32 300	31 300	15	21	20	19	82	76	76	71	41	37	39	28	−0.0001	−0.0001	−0.0001
Z	37 700	35 000	33 400	32 200	15	16	16	13	85	78	78	73	43	39	36	32	−0.0001	0.0000	−0.0001
Grand Average	36 900	34 500	32 800	31 700	15	16	15	14	83	76	76	72	39	37	37	28	−0.0001	−0.0001	−0.0001

Mechanical Testing

American Smelting and Refining Co.
Apex Smelting Co.
Bell Telephone Laboratories, Inc.
Bohn Aluminum and Brass Corp.
The Bunker Hill Co.
Canada Bureau of Mines
Consolidated Mining and Smelting Co. of Canada, Ltd.
Frankford Arsenal
The Hoover Co.
National Bureau of Standards
National Lead Co.
The New Jersey Zinc Co.
Western Electric Co.
Westinghouse Air Brake Co.

Exposure Services

Bell Telephone Laboratories, Inc.
Pennsylvania Railroad
Pennsylvania State University
L. B. Sartain
University of Arizona

Respectfully submitted on behalf of the subcommittee,

D. H. KLEPPINGER, *Chairman*
GEORGE L. WERLEY

REPORT OF COMMITTEE B-7
ON
LIGHT METALS AND ALLOYS, CAST AND WROUGHT*

Committee B-7 on Light Metals and Alloys, Cast and Wrought, held two meetings during the year: on June 30, 1960, in Atlantic City, N. J., in conjunction with the Annual Meeting of the Society; and on December 8, 1960, in Washington, D. C. The subcommittees and the Advisory Committee met prior to the committee meetings.

The committee consists of 98 members, all of whom are voting members; 36 are classified as producers, 39 as consumers, and 23 as general interest members.

Paul P. Mozley and Gordon C. Mutch, who have been members of the committee since 1945 and 1954 respectively, resigned because of retirement or pending retirement.

Appointment of the following representatives to other ASTM committees was made: G. W. Stickley to Subcommittee 6 and Subcommittee 30 of Committee E-1 on Methods of Testing to replace F. M. Howell; L. H. Adam to Subcommittee 14 of Committee E-1 to replace A. Mendizza; and I. V. Williams to the Council of the Division of Material Sciences.

The Advisory Committee continued to function as advisor to the American Standards Assn. in its representation of the United States as a participating member of Technical Committee 79 on Light Metals and Their Alloys of the International Organization for Standardization.[1]

RECOMMENDATIONS AFFECTING STANDARDS

The committee recommends one new specification for publication as tentative, revisions in one tentative specification and its continuation as tentative, continuation of one tentative without revision, adoption of one tentative as standard without revision, adoption of 15 tentatives as standard with revisions, and revision of 7 standards for immediate adoption. The standards and tentatives affected, together with the revisions recommended, are covered in detail in Appendix I to this report.[2]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Aluminum and Aluminum Alloy Ingots (D. L. LaVelle, chairman) revised the Standard Specification for Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 – 60) to delete reference to determining electrical resistivity or conductivity on specimens machined from the ingot, as shown in Appendix I to this report. A task group is developing recommendations for revisions in chemical composition limits and addition of more alloys to the specification.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] The following ISO Recommendations have been accepted by the ISO Council for publication during the past year:
- R164—Composition of Aluminum Alloy Castings,
- R190—Tensile Testing of Light Metals and Their Alloys,
- R191—Brinell Hardness Test for Light Metals and Their Alloys, and
- R192—Diamond Pyramid Hardness Test for Light Metals and Their Alloys.

[2] See p. 286.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee II on Aluminum Alloy Castings (D. L. Colwell, chairman) recommends that the Tentative Specification for Aluminum-Base Alloy Permanent Mold Castings (B 108 – 59 T) be continued as tentative since a revision is being developed to add tensile requirements for test bars machined from castings of alloys SC51B and SG70B.

The subcommittee is cooperating with Subcommittee I in developing revisions in chemical composition limits and addition of more alloys to the Tentative Specification for Aluminum-Base Alloy Sand Castings (B 26 – 60 T) and the Tentative Specification for Aluminum-Base Alloy Permanent Mold Castings (B 108 – 59 T), and has requested the Die Casting and Permanent Mold Division of the American Foundrymen's Society to attempt to develop a standard design for a permanent mold for aluminum alloy test bars.

Subcommittee III on Wrought Aluminum and Wrought Aluminum Alloys (H. D. Monsch, chairman) developed the new Tentative Specification for Aluminum Foil for Capacitors appended to this report.[4]

The subcommittee revised the specifications under its jurisdiction to specify the minimum size of subsize tension test specimens; to add new alloys and tempers; and to revise chemical requirements, tensile requirements, and dimensional tolerances to bring them into agreement with the latest industry practice. In addition, the Tentative Specification for Aluminum-Alloy Drawn Seamless Tubes for Condensers and Heat-Exchangers (B 234 – 60 T) was revised with the assistance of representatives from the Heat Exchange Inst. to specify dimensional tolerances needed for the intended use of the product, to add expansion test requirements, and to provide for deburring of tube ends when desired by the purchaser. The revisions are included in Appendix I to this report.

Subcommittee IV on Magnesium and Magnesium Alloys, Cast and Wrought (A. A. Moore, chairman) revised the Standard Specification for Magnesium-Base Alloy Sand Castings (B 80 – 59) to add alloy QE22A and to increase tensile yield strength requirements on other alloys; revised the Standard Specification for Magnesium-Base Alloy Sheet and Plate (B 90 – 60) to bring the tensile requirements and dimensional tolerances into agreement with latest industry practice; revised the Standard Specification for Magnesium Ingot and Stick for Remelting (B 92 – 60) to add 9990B magnesium, which is used as a reducing agent in production of nuclear grade metals; and revised the Standard Specification for Magnesium-Base Alloy Bars, Rods, and Shapes (B 107 – 60) to change the temper designation from T51 to T5 for alloy AZ80A, and to revise angularity tolerances. The revisions are included in Appendix I to this report.

Subcommittee V on Testing Light Metals (J. C. Millson, chairman) is cooperating with Subcommittee 4 on Tension Testing of Committee E-1 on Methods of Testing (*1*) in establishing equivalence of subsize flat tension test specimens with the standard flat specimen shown in Fig. 7 of Methods of Tension Testing of Metallic Materials (E 8 – 57 T), and in establishing standard sizes for subsize flat specimens; (*2*) in resolving discrepancies between Method E 8 and Federal Test Method Standard No. 151; and (*3*) in making these standards more specific concerning specimens for testing light metals. The subcommittee is also reviewing sampling requirements in ASTM and Government specifications for light metals in preparation for making recommendations for achieving greater uniformity in the sampling requirements.

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[4] The new tentative appears in the 1961 Book of ASTM Standards, Part 2.

Subcommittee VII on Codification of Light Metals and Alloys, Cast and Wrought (C. B. Gleason, chairman) revised the Recommended Practice for Codification of Light Metals and Alloys, Cast and Wrought (B 275 – 59) to change its scope to exclude wrought aluminum and its alloys, to add examples explaining the codification practice, and to specify standard methods for expressing chemical composition limits for aluminum and magnesium and their alloys; and revised the Recommended Practice for Temper Designation of Light Metals and Alloys, Cast and Wrought (B 296 – 56) to amplify it and to define some recently developed tempers. The revisions are included in Appendix I to this report. The subcommittee also compiled the list of alloy designations assigned in conformance with Recommended Practice B 275, and the ASTM specifications in which the alloys appear, which is Appendix II to this report.[5]

Subcommittee VIII on Atmospheric Exposure Tests (L. H. Adam, chairman) has test specimens of 27 aluminum and 8 magnesium commercial alloys, cast and wrought, at five ASTM atmospheric exposure test sites: on top of the Port Authority Building, New York City, for industrial atmospheric conditions; at State College, Pa., for rural; at Kure Beach, N. C., for Atlantic Coast marine; at Point Reyes, Calif., for Pacific Coast marine; and at Freeport, Tex., for Gulf Coast marine atmospheric conditions. Data have been published in ASTM *Proceedings*[6] for specimens exposed six months, one year, and three years, and for cast specimens exposed five years. Data obtained from wrought aluminum and magnesium alloys exposed for five years are given in Appendix III to this report.[7]

Subcommittee IX, Editorial (J. J. Martin, chairman) is preparing Recommendations on Form of ASTM Committee B-7 Specifications to supplement Proposed Recommendations on Form of ASTM Specifications, April, 1957, which have been approved as a guide, insofar as practical, for writing and revising B-7 specifications. Standardization of section titles, sequence, and wording, and form, titles, and headings of tables is being attempted.

Special Subcommittee on Light Metals for Nuclear Applications (W. G. Groth, chairman) cooperated with Subcommittee IV in revising the Standard Specification for Magnesium Ingot and Stick for Remelting (B 92 – 60) to add high-purity magnesium 9990B for use as a reducing agent in the production of nuclear-grade metals, as shown in Appendix I to this report. The subcommittee has not made recommendations for addition of special alloys to the specifications for aluminum products because the standard alloys covered by the specifications are considered suitable for nuclear applications.

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[5] See p. 300.

[6] Data for 6-month and 1-yr exposures: L. H. Adam and M. Dougherty, "Atmospheric Exposure of Wrought Aluminum and Magnesium Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 55, p. 284 (1955); L. H. Adam, "Atmospheric Exposure of Aluminum and Magnesium Sand and Permanent Mold Castings," *Proceedings*, Am. Soc. Testing Mats., Vol. 54, p. 270 (1954).

Data for 3-yr exposures: L. H. Adam and M. Dougherty, "Atmospheric Exposure of Aluminum and Magnesium Sand and Permanent Mold Castings," *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 194 (1958); L. H. Adam and Marie Dougherty, "Atmospheric Exposure of Wrought Aluminum and Magnesium Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 59, p. 289 (1959).

Data for 5-yr exposures: L. H. Adam and M. Dougherty, "Atmospheric Exposure of Aluminum and Magnesium Sand and Permanent Mold Castings," *Proceedings*, Am. Soc. Testing Mats., Vol. 60, p. 186 (1960).

[7] See p. 302.

This report has been submitted to letter ballot of the committee, which consists of 98 members; 83 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. A. HARRIS,
Chairman.

R. B. SMITH,
Secretary.

APPENDIX I

PROPOSED RECOMMENDATIONS AFFECTING STANDARDS ON LIGHT METALS AND ALLOYS, CAST AND WROUGHT

In this Appendix are given the recommendations affecting certain standards and tentatives covering light metals and alloys which are referred to earlier in this report. The standards and tentatives appear in their present form in the 1958 Book of ASTM Standards, Part 2, or in the 1959 or 1960 Supplements to Book of ASTM Standards, Part 2.

NEW TENTATIVE

The committee recommends for publication as tentative the Specification for Aluminum Foil for Capacitors as appended hereto.[1]

REVISION OF TENTATIVE

The committee recommends revisions as follows of the Tentative Specification for Factory Made Wrought Aluminum and Aluminum-Alloy Welding Fittings (**B 361 – 60 T**) and continuation of the specification as tentative:

Section 8(a).—Change to read as follows:

> 8. (*a*) The sizes, shapes and dimensions, and, unless otherwise specified, the end preparation (Note 2), shall be as specified for fittings covered by the American Standard for Steel Butt Welding Fittings (ASA No.: B16.9) and the American Standard for Steel Socket Welding Fittings (ASA No.: B16.11).

New Note 2.—Add the following New Note 2 after Section 8(*a*), renumbering the present Notes 2 and 3 accordingly:

> NOTE 2.—Other end preparations have been developed which appear to be superior to the straight bevel, such as "V" groove of the American Standards (ASA No.: B16.9 and No.: B16.11), under many circumstances. They may be specified when desired. The so-called "modified vee" end preparation described in the American Welding Society document "Recommended Practices for Gas Shielded-Arc Welding of Aluminum and Aluminum Alloy Pipe (AWS D10.7–60) is one example.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Specification for Aluminum-Alloy Drawn Seamless Coiled Tubes for Special Purpose Applications (**B 307 – 60 T**) be approved for reference to letter ballot of the Society for adoption as standard without revision.

ADOPTION OF TENTATIVES AS STANDARD WITH REVISION

The committee recommends that the following 15 tentatives be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

Tentative Specification for Aluminum-Alloy Sheet and Plate (B 209 – 60 T):

Section 7.—Change the second sentence to read: "Requirements for yield strength are included for such applications as pressure vessels, but for most applications involving the use of alloys 1060, 1100, 5005, 5050, 5052, 5652, 5154, 5254, 3003, Alclad 3003, 3004, and Alclad 3004, yield strength is not important."

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[1] The new tentative appears in the 1961 Book of ASTM Standards, Part 2.

Table II.—Delete footnote *h* from the minimum and maximum yield strength values for the O, H32, H34, and H113 tempers of alloy 5083; the 0, H32, H34, H36, and H112 tempers of alloy 5086; the O, H32, H34, and H112 tempers of alloy 5454; and the 0, H24, and H321 tempers of alloy 5456.

Section 9.—Change the second and succeeding sentences to read as follows:

For sheet and plate less than 0.500 in. in thickness, the standard sheet-type specimen shown in Fig. 7 of the Methods of Tension Testing of Metallic Materials (ASTM Designation: E 8) shall be used. For plate 0.500 in. or over in thickness, the standard specimen ½ in. in diameter shown in Fig. 8 of Methods E 8 shall be used, unless the size of the material makes this impracticable. In such cases, round specimens of smaller size proportional to the standard specimen shown in Fig. 8 of Methods E 8 may be used provided that in no case shall the dimensions of the smallest specimen be less than the following:

Diameter of reduced section, in.	¼
Length of reduced section, in.	1¼
Radius of fillet, in.	¼
Diameter of end section, in.	⅜
Over-all length, in.:	
With threaded or shouldered ends	3
With plain cylindrical ends	4

Tentative Specification for Aluminum-Alloy Drawn Seamless Tubes (B 201 – 60 T):

Table I.—Change the maximum zinc limit for alloys 5052 and 5652 from "0.20" to "0.10." Change the note referenced for the iron and silicon limits for 5652 from "*c*" to "*f*" and add the following new footnotes:

[e] Iron plus silicon shall not exceed 1.0 per cent.

[f] Iron plus silicon shall not exceed 0.40 per cent.

Add alloy 1100 with the following chemical requirements:

Aluminum, per cent	99.00 min[b]
Copper, per cent	0.20
Iron, per cent	[e]
Silicon, per cent	[e]
Manganese, per cent	0.05
Zinc, per cent	0.10
Other elements, per cent:	
Each	0.05
Total	0.15

Table II.—Add the alloys, tempers, and tensile requirements shown in the accompanying Table I.

Section 8.—Revise to read:

8. Tension test specimens from round tubes 2 in. or less in outside diameter and from square tubes 1½ in. or less on a side shall be of the full section of the tube unless the limitations of the testing machine preclude the use of such a

TABLE I.—TENSILE REQUIREMENTS.
(Additions to Table II of Specification B 210 – 60 T)

Alloy	Temper	Wall Thickness, in.	Tensile Strength, psi		Yield Strength, min, psi[a]	Elongation in 2 in. or 4 × Diameter, min, per cent[f]	
			Maximum	Minimum		Full-Section Specimen	Cut-out Specimen
1100	0	0.014 to 0.500	15 500	...	...	..	..
	H12	0.014 to 0.500	...	14 000	...	..	..
	H14	0.014 to 0.500	...	16 000	...	..	..
	H16	0.014 to 0.500	...	19 000	...	..	..
	H18	0.014 to 0.500	...	22 000	...	..	..
5652	H32	0.018 to 0.450	...	31 000	23 000[c]	..	..
	H36	0.018 to 0.450	...	37 000	29 000[c]	..	..
5050	H32	0.010 to 0.500	...	22 000	16 000[c]	..	..
5052	H32	0.010 to 0.450	...	31 000	23 000[c]	..	..
3003	H12	0.010 to 0.500	...	17 000	12 000[c]	..	..

APPENDIX I

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In this Appendix are given the recommendations affecting certain standards and tentatives covering light metals and alloys which are referred to earlier in this report. The standards and tentatives appear in their present form in the 1958 Book of ASTM Standards, Part 2, or in the 1959 or 1960 Supplements to Book of ASTM Standards, Part 2.

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The committee recommends revisions as follows of the Tentative Specification for Factory Made Wrought Aluminum and Aluminum-Alloy Welding Fittings (**B 361 – 60 T**) and continuation of the specification as tentative:

Section 8(a).—Change to read as follows:

> 8. (*a*) The sizes, shapes and dimensions, and, unless otherwise specified, the end preparation (Note 2), shall be as specified for fittings covered by the American Standard for Steel Butt Welding Fittings (ASA No.: B16.9) and the American Standard for Steel Socket Welding Fittings (ASA No.: B16.11).

New Note 2.—Add the following New Note 2 after Section 8(*a*), renumbering the present Notes 2 and 3 accordingly:

> NOTE 2.—Other end preparations have been developed which appear to be superior to the straight bevel, such as "V" groove of the American Standards (ASA No.: B16.9 and No.: B16.11), under many circumstances. They may be specified when desired. The so-called "modified vee" end preparation described in the American Welding Society document "Recommended Practices for Gas Shielded-Arc Welding of Aluminum and Aluminum Alloy Pipe (AWS D10.7–60) is one example.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Specification for Aluminum-Alloy Drawn Seamless Coiled Tubes for Special Purpose Applications (**B 307 – 60 T**) be approved for reference to letter ballot of the Society for adoption as standard without revision.

ADOPTION OF TENTATIVES AS STANDARD WITH REVISION

The committee recommends that the following 15 tentatives be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

Tentative Specification for Aluminum-Alloy Sheet and Plate (B 209 – 60 T):

Section 7.—Change the second sentence to read: "Requirements for yield strength are included for such applications as pressure vessels, but for most applications involving the use of alloys 1060, 1100, 5005, 5050, 5052, 5652, 5154, 5254, 3003, Alclad 3003, 3004, and Alclad 3004, yield strength is not important."

---

[1] The new tentative appears in the 1961 Book of ASTM Standards, Part 2.

Table II.—Delete footnote *h* from the minimum and maximum yield strength values for the O, H32, H34, and H113 tempers of alloy 5083; the O, H32, H34, H36, and H112 tempers of alloy 5086; the O, H32, H34, and H112 tempers of alloy 5454; and the O, H24, and H321 tempers of alloy 5456.

Section 9.—Change the second and succeeding sentences to read as follows:

> For sheet and plate less than 0.500 in. in thickness, the standard sheet-type specimen shown in Fig. 7 of the Methods of Tension Testing of Metallic Materials (ASTM Designation: E 8) shall be used. For plate 0.500 in. or over in thickness, the standard specimen ½ in. in diameter shown in Fig. 8 of Methods E 8 shall be used, unless the size of the material makes this impracticable. In such cases, round specimens of smaller size proportional to the standard specimen shown in Fig. 8 of Methods E 8 may be used provided that in no case shall the dimensions of the smallest specimen be less than the following:
>
> | | |
> |---|---|
> | Diameter of reduced section, in. | ¼ |
> | Length of reduced section, in. | 1¼ |
> | Radius of fillet, in. | ¼ |
> | Diameter of end section, in. | ⅜ |
> | Over-all length, in.: | |
> | With threaded or shouldered ends | 3 |
> | With plain cylindrical ends | 4 |

Tentative Specification for Aluminum-Alloy Drawn Seamless Tubes (B 201 - 60 T):

Table I.—Change the maximum zinc limit for alloys 5052 and 5652 from "0.20" to "0.10." Change the note referenced for the iron and silicon limits for 5652 from "*c*" to "*f*" and add the following new footnotes:

> [e] Iron plus silicon shall not exceed 1.0 per cent.
>
> [f] Iron plus silicon shall not exceed 0.40 per cent.

Add alloy 1100 with the following chemical requirements:

Aluminum, per cent	99.00 min[b]
Copper, per cent	0.20
Iron, per cent	[e]
Silicon, per cent	[e]
Manganese, per cent	0.05
Zinc, per cent	0.10
Other elements, per cent:	
Each	0.05
Total	0.15

Table II.—Add the alloys, tempers, and tensile requirements shown in the accompanying Table I.

Section 8.—Revise to read:

> 8. Tension test specimens from round tubes 2 in. or less in outside diameter and from square tubes 1½ in. or less on a side shall be of the full section of the tube unless the limitations of the testing machine preclude the use of such a

TABLE I.—TENSILE REQUIREMENTS.
(Additions to Table II of Specification B 210 - 60 T)

Alloy	Temper	Wall Thickness, in.	Tensile Strength, psi		Yield Strength, min, psi[d]	Elongation in 2 in. or 4 × Diameter, min, per cent[f]	
			Maximum	Minimum		Full-Section Specimen	Cut-out Specimen
1100	O	0.014 to 0.500	15 500	...	...	..	..
	H12	0.014 to 0.500	...	14 000	...	..	..
	H14	0.014 to 0.500	...	16 000	...	..	..
	H16	0.014 to 0.500	...	19 000	...	..	..
	H18	0.014 to 0.500	...	22 000	...	..	..
5652	H32	0.018 to 0.450	...	31 000	23 000[c]	..	..
	H36	0.018 to 0.450	...	37 000	29 000[c]	..	..
5050	H32	0.010 to 0.500	...	22 000	16 000[c]	..	..
5052	H32	0.010 to 0.450	...	31 000	23 000[c]	..	..
3003	H12	0.010 to 0.500	...	17 000	12 000[c]	..	..

specimen. For tubes of larger size, or when it is not possible to test the full section, the standard specimens shown in Fig. 7 or Fig. 8 or as specimen No. 1 of Fig. 13 of the Methods of Tension Testing of Metallic Materials (ASTM Designation: E 8) may be used. When the size of the material makes it impracticable to use any of these specimens, round specimens proportional to the standard specimen shown in Fig. 8 of Methods E 8 may be used provided that in no case shall the dimensions of the smallest specimen be less than the following:

Diameter of reduced section, in.	¼
Length of reduced section, in.	1¼
Radius of fillet, in.	¼
Diameter of end section, in.	⅜
Over-all length, in.:	
With threaded or shouldered ends	3
With plain cylindrical ends	4

Table III.—Add reference to alloy 1100 in Columns 2 and 3.

Table IV.—Add reference to alloy 1100 in Columns 2 and 3.

Table V.—Delete Footnote *a*, and reference thereto. Change the heading of the second column to read: "At Corners, Square and Rectangular." Revise the size ranges in the first column to read: "0.500 and less, 0.501 to 1.000, 1.001 to 2.000, 2.001 to 3.000, 3.001 to 5.000, 5.001 to 6.000, 6.001 to 8.000, 8.001 to 10.000, over 10.000."

New Table.—Add a new Table VI to read as shown in the accompanying Table II,[2] and renumber present Table VI and succeeding tables accordingly.

Section 13.—Revise to read: "Variation from the specified or nominal dimensions shall not exceed the amounts prescribed in Tables III, IV, V, VI, VII, VIII, IX, X, XI, XII, and XIII."

Tentative Specification for Aluminum-Alloy Bars, Rods, and Wire (B 211–60 T):

Table I.—For alloy 5652, change the footnote reference "*c*" appearing in Columns 4 and 5 to read "*g*."

Add a new Footnote *g* at the bottom of the table to read, "[g]The permissible maximum amount of iron plus silicon is 0.40 per cent."

For alloys 5052 and 5652, change the maximum zinc percentage from "0.20" to "0.10."

Table II.—In Column 2 after the word "Temper" add reference to Footnote *i*.

In second column for alloy 2024 revise "T4" to read "T4 and T351[h]."

For alloy 2017 revise "T4" to read "T4 and T451[h]."

For alloy 2014 revise "T4" to read "T4 and T451" and revise "T6" to read "T6 and T651[h]."

For alloy 6061 revise "T4" to read "T4 and T451" and revise "T6" to read "T6 and T651[h]."

For alloy 7075 revise "T6" to read "T6 and T651[h]."

For alloy and temper 2024-T4 and T351 change the elongation percentage for thickness 0.125 to 6.500[e] from "14" to "10."

For alloy and temper 2017-T4 and T451 change the elongation percentage for thickness 0.125 to 8.000[f] from "16" to "12."

For alloy and temper 2014-T4 and T451 change the thickness increment now reading "0.125 to 6.750[e]" to read "0.125 to 8.000[e]."

For alloy and temper 2014-T6 and T651 change the thickness increment now reading "0.125 to 6.750[e]" to read "0.125 to 6.500[e]."

Add a new Footnote *h* at bottom of table to read: "[h] For stress-relieved tempers, characteristics and properties other than those specified may differ somewhat from the corresponding characteristics and properties of material in the basic tempers."

Add a new Footnote *i* at bottom of table to read: "[i]For explanation of temper designation, see Recommended

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[2] The new table is not included in this report but appears in the revised specification; see 1961 Book of ASTM Standards, Part 2.

Practice for Temper Designation of Light Metals and Alloys, Cast and Wrought (ASTM Designation: B 296)."

Add alloys, tempers, and tensile requirements as shown in the accompanying Table III.[2]

Section 8(a).—Revise to read as follows:

"Tension test specimens shall be the full section of the material, or specimens machined from it. Machined specimens may be the standard sheet-type specimen shown in Fig. 7 of the Methods of Tension Testing of Metallic Materials (ASTM Designation: E 8) or the standard specimen ½ in. in diameter shown in Fig. 8 of Methods E 8. If the size and shape of the material makes it impracticable to use any of these specimens, then round specimens of smaller size proportional to the standard specimen shown in Fig. 8 of Methods E 8 may be used, provided that in no case shall the dimensions of the smallest specimen be less than the following:

Diameter of reduced section, in.	¼
Length of reduced section, in.	1¼
Radius of fillet, in.	¼
Diameter of end section, in.	⅜
Over-all length, in.:	
With threaded or shouldered ends	3
With plain cylindrical ends	4

Section 12.—Change the heading to read "Dimensional Tolerances," and revise the Section to read:

12. Variations from specified dimensions for the class of material ordered shall not exceed the amounts specified in Tables III, IV, V, VI, and VII as applicable. The degrees of twist, straightness, deviation from flat (flat surfaces) angularity, and squareness of cut ends shall not exceed the amounts specified in Tables VIII, IX, X, XI, XII, XIII, and XIV.

TABLE XIII.—CHEMICAL REQUIREMENTS.
(Additions to Table I, Specification B 221 - 60 T)

Alloy	6351	7079	7178
Aluminum, per cent	remainder	remainder	remainder
Copper, per cent	0.10	0.40 to 0.8	1.6 to 2.4
Iron, per cent	0.50	0.40	0.7
Silicon, per cent	0.7 to 1.3	0.30	0.50
Manganese, per cent	0.40 to 0.8	0.10 to 0.30	0.30
Magnesium, per cent	0.40 to 0.8	2.9 to 3.7	2.4 to 3.1
Zinc, per cent	...	3.8 to 4.8	6.3 to 7.3
Chromium, per cent	...	0.10 to 0.25	0.18 to 0.40
Titanium, per cent	0.20	0.10	0.20
Other elements, per cent:			
Each	0.05	0.05	0.05
Total	0.15	0.15	0.15

Table III.—Revise to read as shown in the accompanying Table IV,[2] including the revisions in the title and headings.

Table IV.—Replace with the accompanying Table V.[2]

Table V.—Delete and replace with a new table as shown in the accompanying Table VI.[2]

New Table.—Add new Table VI as shown in the accompanying Table VII.[2]

Table VI.—Renumber as Table VII.

Table VII.—Renumber as Table XI and change the title to read, "Permissible Variations from Straightness of Rolled or Cold-Finished Rod and Bar in All Tempers Except O, T351, T451, T651."

Table VIII.—Revise to read: "Allowable deviation from square: 1 deg."

New Tables.—Add new Tables IX, X, XII, XIII, XIV as shown in the accompanying Tables VIII, IX, X, XI, XII.[2]

Tentative Specification for Aluminum-Alloy Extruded Bars, Rods, and Shapes (B 221 - 60 T):

Table I.—Delete the chemical requirements for alloys 5254 and 6053 and add chemical requirements for alloys 6351, 7079, and 7178 as shown in the accompanying Table XIII. Change per cent aluminum for alloy 1100 to read: "99.00." Delete column headed "Beryllium."

Table II.—Delete the tensile requirements for alloys 5254 and 6053, add tensile requirements for alloys 6351, 7079, and 7178 and change to read as shown in the accompanying Table XIV.[2]

Section 7.—Change the second sentence to read: "Requirements for yield strength are included for such applications as pressure vessels, but for most applications involving the use of alloys 1100, 3003, and 5154, yield strength is not important."

Section 8(a).—Change to read as follows:

> 8. (*a*) Tension test specimens shall be the full section of the material, or specimens machined from it. Machined specimens may be the standard sheet-type specimen shown in Fig. 7 of the Methods of Tension Testing of Metallic Materials (ASTM Designation: E 8) or the standard ½-in. diameter specimen shown in Fig. 8 of Methods E 8. If the size and shape of the material make it impracticable to use any of these specimens, then round specimens of smaller size proportional to the standard specimen shown in Fig. 8 of Methods E 8 may be used, provided that in no case shall the dimensions of the smallest specimen be less than the following:

Diameter of reduced section, in.	¼
Length of reduced section, in.	1¼
Radius of fillet, in.	¼
Diameter of end section, in.	⅜
Over-all length, in.:	
With threaded or shouldered ends	3
With plain cylindrical ends	4

Section 12.—Change the heading to "Dimensional Tolerances" and revise the section to read: "Variations from the specified dimensions for the class or type of materials ordered shall not exceed the amounts specified in Tables III, IV, V, VI, VII, VIII, IX, X, XI, XII, XIII, XIV, and XV."

New Tables.—Add new Tables III through XV as shown in the accompanying Tables XV through XXVII.[2]

Tentative Specification for Aluminum-Alloy Drawn Seamless Tubes for Condensers and Heat Exchangers (B 234 - 60 T):

Section 1.—Change to read: "This specification covers aluminum-alloy drawn seamless tubes, designated as shown in Table I, for use in surface condensers, evaporators, and heat exchangers."

Section 2.—Insert the following new Item *7* and renumber present Items *7* and *8* accordingly: "(*7*) Whether cut ends of the tube are to be deburred (Section 16(*b*))."

Section 5(a).—Change the first sentence to read as follows: "Samples for chemical analysis of alloys 1060, 3003, 5052, 5454, 6061, and 6062 shall be taken in accordance with the Method of Sampling Wrought Non-Ferrous Metals and Alloys for Determination of Chemical Composition (ASTM Designation: E 55), except that the weight of the prepared sample may be a minimum of 75 g."

Table I.—Add alloys 5052 and 5454 with the following chemical requirements:

	5052	5454
Aluminum, per cent	remainder	remainder
Copper, per cent	0.10	0.10
Iron, per cent	*c*	*d*
Silicon, per cent	*c*	*d*
Manganese, per cent	0.10	0.50 to 1.0
Magnesium, per cent	2.2 to 2.8	2.4 to 3.0
Zinc, per cent	0.10	0.25
Chromium, per cent	0.15 to 0.35	0.05 to 0.20
Titanium, per cent	...	0.20
Other elements, per cent:		
Each	0.05	0.05
Total	0.15	0.15

Change reference to Footnote *c* after

the aluminum limit for 1060 to read "*e*." Delete the present Footnote *c*, and add the following new footnotes:

[c] Iron plus silicon shall not exceed 0.45 per cent.

[d] Iron plus silicon shall not exceed 0.40 per cent.

[e] The aluminum content for unalloyed aluminum not made by a refining process is the difference between 100.00 per cent and the sum of all other metallic elements present in amounts of 0.010 per cent or more each, expressed to the second decimal.

Table II.—Add the alloys, tempers, and tensile requirements shown in the accompanying Table XXVIII. Change the wall thickness increment for 6061-T4, -T6 and for 6062-T4, -T6 from "0.050 to 0.200" to read "0.050 to 0.249." Add the following new Footnote *c* and reference it in the heading of the second column:

[c] These temper designations were established in accordance with the Recommended Practice for Temper Designation of Light Metals and Alloys, Cast and Wrought (ASTM Designation: B 296).

Section 8.—Change to read as follows:

8. Tension test specimens shall be of the full section of the tube unless the limitations of the testing machine preclude the use of such a specimen. When it is not possible to test the full section, the standard specimen shown in Fig. 7 or specimen No. 1 of Fig. 13 of the Methods of Tension Testing of Metallic Materials (ASTM Designation: E 8) may be used. When the size of the material makes it impracticable to use any of these specimens, round specimens proportional to the standard specimen shown in Fig. 8 of Methods E 8 may be used provided that in no case shall the dimensions of the smallest specimen be less than the following:

Diameter of reduced section, in.	¼
Length of reduced section, in.	1¼
Radius of fillet, in.	¼
Diameter of end section, in.	⅜
Over-all length, in.:	
With threaded or shouldered ends	3
With plain cylindrical ends	4

Section 13.—Delete and replace with the following new section with the heading "Expansion Test:"

13. The tube ends shall be capable of being flared by driving a steel pin having a taper of 1½ in. per ft into the tube until the inside diameter has been increased 20 per cent. The expanded tube end shall show no cracking or rupture visible to the unaided eye.

Section 15(a).—Revise the first sentence to read as follows:

When measured by use of "go" and "no-go" ring gages, the outside diameter of 1060, 3003, Alclad 3003, 5052, and 5454 alloy tubes shall not vary from that specified by more than the amounts prescribed in Table IV, and the outside diameter of tubes in alloys 6061 and 6062 shall not vary from that specified by more than amounts prescribed in Table III.

Section 15(b).—Change reference to Table IV to read "Table V."

Section 15(c).—Change reference to Table V to read "Table VI."

Section 15(d).—Change reference to Table VI to read "Table VII."

Table III.—Change the title to read: "Permissible Variations in Diameter of 6061 and 6062 Alloy Tubes."

New Table.—Add a new Table IV as shown in the accompanying Table XXIX.

Table IV.—Renumber as Table V.

Table V.—Renumber as Table VI and revise to read as shown in the accompanying Table XXX.[2]

Table VI.—Renumber as Table VII and change the diameter range in the first column to read: "0.375 to 2.000, incl."

Section 16.—Change to read as follows:

16. (*a*) Unless otherwise agreed upon by the manufacturer and the purchaser, tubes shall be supplied in the mill finish and shall be of uniform quality and temper, clean, sound, and free from injurious defects.

(*b*) When so specified on the purchase order, the cut ends of the tubes shall be deburred by use of a wire wheel, file, or other suitable tool or device.

Appendix I.—Change the second sentence to read: "Alloys 1060, 3003, 5052, and 5454 are supplied in a strain-hardened temper to meet the specified tensile and yield strengths."

Tentative Specification for Aluminum-Alloy Extruded Tubes (B 235 - 60 T):

Revise as appended hereto.[3]

Tentative Specification for Aluminum Bars for Electrical Purposes (Bus Bars) (B 236 - 59 T):

Section 6.—Add the following note:

NOTE.—For purposes of determining conformance with this specification, each value for electrical resistivity shall be rounded off to the nearest unit in the last right-hand place of figures, in accordance with the rounding-off method of the Recommended Practices for Designating Significant Places in Specified Limiting Values (ASTM Designation: E 29).

Section 14.—Delete the section and renumber subsequent sections accordingly.

Tentative Specification for Aluminum-Alloy Pipe (B 241 - 60 T):

Table I.—Revise title and table format and add requirements for alloys 5154, 5254, and 6351 as shown in the accompanying Table XXXI.[2]

Section 7.—Change the heading to read "Specified Mechanical Properties," and change the section to read as follows:

7. Pipe shall be supplied in the alloy and temper specified in the contract or purchase order and shall conform to the requirements as to tensile properties prescribed in Table II. Requirements for yield strength are included for such applications as pressure vessels, but for most applications involving the use of alloys 3003, 5154, and 5254, yield strength is not important. For these alloys, yield strength need not be determined, unless specifically indicated in the contract or purchase order.

Table II.—Revise table title and format, and add requirements for alloys 5154, 5254, and 6351 as shown in the accompanying Table XXXII.[2]

Table III.—Revise table title, format and coverage as shown in the accompanying Table XXXIII.[2]

Section 8.—Change to read as follows:

8. Tension test specimens of pipes 1½ in. or less in size shall be a full section of the pipe unless the limitations of the testing machine preclude the use of such a specimen. For pipe sizes larger than 1½ in., or when it is not possible to test the full section, the standard specimens shown in Fig. 7 or Fig. 8 or specimen No. 1 of Fig. 13 of the Methods of Tension Testing of Metallic Materials (ASTM Designation: E 8) may be used. When the size of the material makes it impractical to use any of these specimens, round specimens proportional to the standard specimen shown in Fig. 8 of Methods E 8 may be used provided that in no case shall the dimensions of the smallest specimen be less than the following:

Diameter of reduced section, in.	¼
Length of reduced section, in.	1¼
Radius of fillet, in.	¼
Diameter of end section, in.	⅜
Over-all length, in.:	
With threaded or shouldered ends	3
With plain cylindrical ends	4

Section 12.—Change the heading to read "Tolerances Applicable to Dimensions and Weight," and change the Section to read as follows:

12. (*a*) *Diameter.*—Outside diameter of pipe shall not vary from nominal outside diameter by an amount greater than that shown in Table IV. Specific minimum and maximum outside diameters are shown in Table III.

(*b*) *Wall Thickness.*—Wall thickness shall not vary from nominal wall thickness by an amount greater than that shown in Table V. For schedules 5 and 10 specific minimum and maximum wall thicknesses are shown in Table III. For schedules 20 and greater, specific minimum wall thicknesses are shown in Table III.

(*c*) *Length.*—Length of individual pieces of pipe shall not vary from that specified by an amount greater than that shown in Table VI.

(*d*) *Weight.*—Weight of pipe shall not vary from theoretical nominal by an amount greater than that specified in Table VII. Specific maximum weights per foot for schedules 20 and greater are shown in Table III.

(*e*) *Straightness.*—The pipe shall not vary from straight by an amount greater than that shown in Table VIII.

Table IV.—Change the table title

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[3] The revised specification appears in the 1961 Book of ASTM Standards, Part 2.

and format as shown in the accompanying XXXIV.[2]

Table V.—Change the table title and format as shown in the accompanying Table XXXV.[2]

New Table.—Add a new Table VI as shown in the accompanying Table XXXVI.[2]

Table VI.—Renumber as Table VII and change the table title and format as shown in the accompanying Table XXXVII.[2]

Table VII.—Renumber as Table VIII and change the table title and format as shown in the accompanying Table XXXVIII.[2]

TABLE XXVIII.—TENSILE REQUIREMENTS.
(Additions to Table II of Specification B 234 - 60 T)

Alloy	Temper[c]	Wall Thickness, in.	Tensile Strength, min, psi	Yield Strength, min, psi[a]	Elongation in 2 in. or 4 × Diameter, min, per cent[b]	
					Full-Section Specimens	Cut-Out Specimens
3003	H25	0.010 to 0.200	22 000	19 000	....	....
Alclad 3003	H25	0.010 to 0.200	21 000	18 000	....	....
5052	H32	0.010 to 0.200	31 000	23 000	....	....
	H34	0.010 to 0.200	34 000	26 000	....	....
5454	H32	0.010 to 0.200	36 000	26 000	....	....
	H34	0.010 to 0.200	39 000	29 000	....	....

Tentative Specification for Aluminum-Alloy Die Forgings (B 247 - 60 T):

Section 9—Change to read as follows:

9. Tension test specimens representing the forgings shall be the standard ½-in. diameter specimen shown in Fig. 8 of the Methods of Tension Testing of Metallic Materials (ASTM Designation: E 8). If the size and shape of the material makes it impracticable to use this specimen, round specimens of smaller size proportional to the standard specimen shown in Fig. 8 of Methods E 8 may be used, provided that in no case shall the dimensions of the smallest specimen be less than the following:

Diameter of reduced section, in.	¼
Length of reduced section, in.	1¼
Radius of fillet, in.	¼
Diameter of end section, in.	⅜
Over-all length, in.:	
With threaded or shouldered ends	3
With plain cylindrical ends	4

The specimens may be machined from a section of the stock used for making the forgings, from a coupon forged from the stock, from a prolongation of the forging, or from one of the forgings in the lot. In all cases, the axis of the test specimen shall be as nearly parallel to the direction of maximum flow of metal as possible. Specimens representing heat-treated forgings shall be heat treated with the forgings they represent or shall be machined from coupons that have been so treated.

Tentative Specification for Aluminum-Alloy Standard Structural Shapes, Rolled or Extruded (B 308 - 60 T):

Table I.—Delete the column for beryllium, and add alloy 6351 with the following chemical requirements:

Aluminum, per cent	remainder
Copper, per cent	0.10
Silicon, per cent	0.7 to 1.3
Iron, per cent	0.50
Manganese, per cent	0.40 to 0.8
Magnesium, per cent	0.40 to 0.8
Titanium, per cent	0.20
Other elements, per cent:	
Each	0.05
Total	0.15

Table II.—Add the alloys, tempers, and tensile requirements as shown in the accompanying Table XXXIX.

Table III.—Change to read as shown in the accompanying Table XL.[2]

Section 8.—Change to read as follows:

8. Tension test specimens shall be machined from the shape with the axis parallel to the length. They may be the standard sheet-type specimens shown in Fig. 7 of the Methods of Tension Testing of Metallic Materials (ASTM Designation: E 8) or the standard ½-in. diameter specimen shown in Fig. 8 thereof, depending on the size of the structural shape. If the size of the material makes it impracticable to use either of these two specimens, round specimens of smaller size proportional to the standard specimen in Fig. 8 of Methods E 8 may be used provided that, in no case shall the dimensions of the smallest specimen be less than the following:

TABLE XXXIX.—TENSILE REQUIREMENTS.
(Additions to Table II of Specification B 308 – 60 T)

Alloy	Temper	Thickness, in.	Area, sq in.	Tensile Strength, min, psi	Yield Strength, min, psi	Elongation in 2 in. or 4 × Diameter, min, per cent
5456......	H111....	5.000 and under	32 and under	44 000	26 000	12
6351......	T4.......	0.749 and under	...	35 000	21 000	17
6351......	T6.......	0.749 and under	...	42 000	37 000	10

Tentative Specification for Aluminum-Alloy Round Welded Tube (B 313 – 60 T):

Table I.—Change the maximum limit for zinc for alloy 5052 from "0.20" to "0.10."

Tentative Specification for Aluminum-Alloy Rivet and Cold Heading Wire and Rods (B 316 – 60 T):

Table I.—Add chemical limits for alloys 5052 and 5652 and new Footnotes *e* and *f* as follows:

	5052	5652
Aluminum.........	remainder	remainder
Copper...........	0.10	0.04
Iron.............	[e]	[f]
Silicon..........	[e]	[f]
Manganese........	0.10	0.01
Magnesium........	2.2 to 2.8	2.2 to 2.8
Zinc.............	0.10	0.10
Titanium.........	...	...
Chromium.........	0.15 to 0.35	0.15 to 0.35
Others:		
Each...........	0.05	0.05
Total..........	0.15	0.15

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[e] Silicon plus iron shall be 0.45 per cent, max.

[f] Silicon plus iron shall be 0.40 per cent, max.

Table II.—Add tensile requirements for alloys 5052 and 5652 as shown in the accompanying Table XLI.

Section 8.—Change to read as follows:

8. (*a*) Tension test specimens shall be the full section of the material, or specimens machined from it. Machined specimens may be the standard ½-in. diameter specimen shown in Fig. 8 of the Methods of Tension Testing of Metallic Materials (ASTM Designation: E 8). If the size of the material makes it impracticable to use either of these two specimens, round specimens of smaller size proportional to the standard specimen shown in Fig. 8 of Methods E 8 may be used, provided that in no case shall the dimensions of the smallest specimen be less than the following:

Diameter of reduced section, in...........	¼
Length of reduced section, in.............	1¼
Radius of fillet, in......................	¼
Diameter of end section, in...............	⅜
Over-all length, in.:	
With threaded or shouldered ends......	3
With plain cylindrical ends...........	4

(*b*) Shear test specimens shall be full cross-sections of the wire up to and including 0.374 in. in diameter except that wire sizes between the standard sizes as shown in Fig. 1 may be turned down to the next smaller jig size. Rod over 0.375 in. in diameter may be turned down to 0.374 in. diameter or smaller for testing.

Tentative Specification for Extruded Aluminum-Alloy Bar, Rod, Pipe, and Structural Shapes for Electrical Purposes (B 317 – 60 T):

Section 13(a).—Change to read as follows:

13. (*a*) *Tension.*—Tension test specimens shall be the full section of the material, or specimens machined from it, with the axis parallel to the length. Machined specimens shall be the standard sheet-type specimen shown in Fig. 7 of the Methods of Tension Testing of Metallic Materials (ASTM Designation: E 8), the specimen shown as No. 1 in Fig. 13 (for pipe only), or the standard ½-in. diameter specimen shown in Fig. 8 thereof. If the size and shape of the material makes it impracticable to use any of these specimens, round specimens of smaller size proportional to the standard specimen shown in Fig. 8 may be used provided that in no case shall the dimensions of the smallest specimen be less than the following:

Diameter of reduced section, in.	¼
Length of reduced section, in.	1¼
Radius of fillet, in.	¼
Diameter of end section, in.	⅜
Over-all length, in.:	
With threaded or shouldered ends	3
With plain cylindrical ends	4

TABLE XLI.—ADDITIONS TO TABLE II, SPECIFICATION B 316 – 60 T.

Alloy	Temper	Diameter,[a] in.	Tensile Strength, psi	
			min	max
5052	O	0.501 and over	...	32 000
	H32	0.500 and under	31 000	37 000
5652	O	0.501 and over	...	32 000
	H32	0.500 and under	31 000	37 000

TABLE XLII.—REVISIONS OF TABLE II, SPECIFICATION B 345 – 60 T.

Alloy	Temper	Pipe Size, in.	Tensile Strength, min, psi	Yield Strength min, psi	Elongation in 2 in. or 4 × Diameter, min, per cent
5083	-H112	All	40 000	...	12
6063	-T42	All (Up through 0.500 wall thickness)	17 000	9 000	12
		All (0.501 to 1.000 wall thickness)	16 000	8 000	12
6063	-T5	All (Up through 0.500 wall thickness)	22 000	16 000	8
		All (0.501 to 1.000 wall thickness)	21 000	15 000	8
6063	-T6	All	30 000	25 000	8
6351	-T4	All	32 000	19 000	16
6351	-T6	All	42 000	37 000	10

Tentative Specification for Type A and Type B Aluminum-Alloy Drawn Annealed Seamless Coiled Tubes (B 318 – 60 T):

Table II.—Change the maximum limit for zinc for alloy 5052 from "0.20" to "0.10."

Section 8.—Change the second sentence to read: "Specimen No. 1 of Fig. 13 of the Methods of Tension Testing of Metallic Materials (ASTM Designation: E 8) may be used in those cases when a full-section specimen cannot be used."

Tentative Specification for Aluminum-Alloy Pipe for Gas and Oil Transmission and Distribution Piping Systems (B 345 – 60 T):

Table I.—Add requirements for alloys

5083 and 6351 as follows:

	5083	6351
Aluminum	remainder	remainder
Copper	0.10	0.10
Iron	0.40	0.50
Silicon	0.40	0.7 to 1.3
Manganese	0.30 to 1.0	0.40 to 0.8
Magnesium	4.0 to 4.9	0.40 to 0.8
Zinc	0.25	...
Chromium	0.05 to 0.25	...
Titanium	0.15	0.20
Other:		
Each	0.05	0.05
Total	0.15	0.15

Table II.—Delete the column "Wall Thickness." Delete requirements for 1060-H18, 3003-H18, 3004-H34, and 5052-H38. Revise requirements for alloy 6063, and add requirements for 5083-H112, 6351-T4, and 6351-T6 as shown in the accompanying Table XLII.

Section 14(a).—Add a new note to the end of this paragraph to read:

> NOTE.—Other end preparations have been developed which appear to be superior to the straight bevel, i.e. "V" groove of Fig. 1 of this specification, under many circumstances. They may be specified when desired. The so-called "modified vee" and preparation described in the American Welding Society document "Recommended Practices for Gas Shielded-Arc Welding of Aluminum and Aluminum Alloy Pipe" (AWS D10.7—60) is one example.

Section 15(c).—Change the last sentence to read: "For specified lengths over 20 ft through 40 ft the allowable variation from specified length shall be plus ½ in."

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following 7 standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

TABLE XLV.—REVISIONS AND ADDITIONS TO TABLE II OF SPECIFICATION B 90 - 60.

Alloy and Condition	Thickness, in.	Tensile Strength, min, psi	Yield Strength (0.2 per cent offset), min, psi	Elongation in 2 in., min, per cent
AZ31B-O	2.001 to 3.000	30 000[a]	...	9
AZ31B-H24	2.001 to 3.000	34 000	18 000	8
HK31A-H24	0.251 to 1.000	34 000	25 000	4
	1.001 to 3.000	33 000	25 000	4
HM21A-T8	0.251 to 0.500	32 000	21 000	6
	0.501 to 1.000	30 000	19 000	6
	1.001 to 2.000	29 000	18 000	6
ZE10A-O	0.016 to 0.060	30 000	18 000	15
	0.061 to 0.250	30 000	15 000	15
	0.251 to 0.500	29 000	12 000	12
ZE10A-H24	0.016 to 0.125	36 000	25 000	4
	0.126 to 0.188	34 000	22 000	4
	0.189 to 0.250	31 000	20 000	4

Standard Specification for Magnesium-Base Alloy Sand Castings (B 80 - 59)

Table I.—Add alloy QE22A with the following chemical requirements:

Magnesium, per cent	remainder
Silver, per cent	2.0 to 3.0
Rare earths, per cent	1.8 to 2.5[b]
Zirconium, per cent	0.40 to 1.0
Copper, max, per cent	0.10
Nickel, max, per cent	0.01
Other impurities, max, per cent	0.30

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[b] Rare earth elements are in the form of didymium.

Table II.—Change to read as shown in the accompanying Table XLIII.[2]

Table III.—Change to read as shown in the accompanying Table XLIV.[2]

New Explanatory Note.—Add the following explanatory note after the one for alloy K1A: "*Alloy QE22A.*—This alloy is primarily used where a high yield strength is needed at temperatures up to 600 F."

Standard Specification for Magnesium-Base Alloy Sheet and Plate (B 90 – 60):

Table II.—For AZ31B-0, AZ31B-H24, and HM21A-T8, add tensile requirements for plate; for HK31A-H24, revise the tensile requirements for plate; and for ZE10A-0 and ZE10A-H24, revise tensile requirements for all thicknesses; as shown in the accompanying Table XLV.

Table III.—Change as shown in the accompanying Table XLVI.[2]

Table VI.—Change as shown in the accompanying Table XLVII.[2]

Table VII. Change as shown in the accompanying Table XLVIII.[2]

Standard Specification for Magnesium Ingot and Stick for Remelting (B 92 – 60):

Section 1.—Change to read: "This specification covers magnesium in the form of ingot and stick for remelting: 9980A is available in ingot and stick form, and 9990A, 9990B, and 9995A are generally available only in ingot form."

Table I.—Add grade 9990B with the following chemical requirements:

Aluminum, max, per cent	0.005
Iron, max, per cent	0.01
Manganese, max, per cent	0.004
Nickel, max, per cent	0.001
Silicon, max, per cent	0.005
Other impurities, each, max, per cent	0.01
Magnesium, by difference, min, per cent	99.90

Standard Specification for Magnesium-Base Alloy Bars, Rods, and Shapes (B 107 – 60):

Table II.—Change AZ80A-T51 to read "AZ80A-T5."

Table VI.—Change as shown in the accompanying Table XLIX.[2]

Table XI.—Change AZ80A-T51 to read "AZ80A-T5."

Standard Specification for Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 – 60):

Section 10.—Change to read as follows:

10. Resistivity or conductivity shall be determined on ingot or on test bars separately cast in permanent molds. The separately-cast test specimens shall be of suitable size and shape appropriate to the instrument and method of test used in making the determination.

Section 12.—Change to read as follows:

12. Electrical resistivity or conductivity shall be determined in accordance with the Method of Test for Resistivity of Electrical Conductor Materials (ASTM Designation: B 193) or the Method of Test for Electrical Conductivity by Use of Eddy Currents (ASTM Designation: B 342), provided that, in case of dispute, the results secured by Method B 193 shall be the basis for acceptance.

Recommended Practice for Codification of Light Metals and Alloys, Cast and Wrought (B 275 – 59):

Section 1.—Change to read as follows:

1. (*a*) This recommended practice covers a system originally adopted for designating light metals and alloys, cast and wrought, and later extended to certain heavier base metal die casting alloys as indicated below. The designations used in the following ASTM Specifications under the jurisdiction of ASTM Committees B-6 on Die-Cast Metals and Alloys and B-7 on Light Metals and Alloys, Cast and Wrought conform to this recommended practice: ASTM Committee B-6 specifications for aluminum-base, copper-base, lead-base, tin-base, and zinc-base alloy die castings, and zinc-base alloy ingot for die castings; ASTM Committee B-7 specifications for aluminum

and aluminum alloy ingot and castings, and magnesium and magnesium alloy ingot, castings, and wrought products.

(*b*) The designations used in ASTM Committee B-7 specifications for aluminum and aluminum alloy wrought products conform to the American Standard Alloy Designation System for Wrought Aluminum (ASA No.: H35.1—1957).

Section 2.—Revise to read as follows, and delete Footnote 3:

2. (*a*) The designations for alloys and unalloyed metals are based on their chemical composition limits (Note 1).

NOTE 1.—For aluminum and magnesium alloys, cast and wrought, standard limits for alloying elements and impurities are expressed to the following places:

Less than 0.0001 per cent (used only for magnesium alloys)	0.0000X
0.0001 to 0.001 per cent	0.000X
0.001 to 0.01 per cent	0.00X
0.01 to 0.1 per cent	
Unalloyed aluminum made by a refining process	0.0XX
Alloys and unalloyed aluminum or magnesium not made by a refining process	0.0X
0.1 through 0.5 per cent	0.XX
Over 0.5 per cent	0.X, X.X, XX.X

(*b*) Designations are assigned, revised, and canceled by Subcommittee VII of ASTM Committee B-7 on Light Metals and Alloys, Cast and Wrought, on written request to its secretary. Complete chemical composition limits shall be submitted with requests for assignment or revision of designations. Arbitrary assignments by other subcommittees or committees will not be recognized.

(*c*) The temper designation, which is used for all metal forms except ingot, follows the alloy designation and is separated therefrom by a dash.

New Appendices.—Add new Appendices I and II to read as follows:

APPENDIX I

EXAMPLES OF CODIFICATION

Example 1.—For alloy C4A in Specifications B 26 and B 179, "C" represents copper which is the only alloying element specified; "4" indicates that the round-off mean percentage of copper lies between 3.5 and 4.5 (actually it is 4.5 and therefore the nearest whole even number is used); and "A" indicates that this is the first alloy qualified and assigned under the designation C4 (see Example 2).

Example 2.—For alloys AZ91A, B, and C, in Specification B 93, "A" represents aluminum, the alloying element specified in the greatest amount; "Z" represents zinc, the alloying element specified in the second greatest amount; "9" indicates that the round-off mean aluminum percentage lies between 8.6 and 9.4; "1" signifies that the rounded-off mean of the zinc lies between 0.6 and 1.4; and "A" as the final letter indicates that this is the first alloy whose composition qualified assignment of the designation AZ91. The final serial letters B and C signify alloys subsequently developed whose specified compositions differ slightly from the first and from one another but do not differ sufficiently to effect a change in the basic designation.

APPENDIX II

Designations assigned in conformance with this Recommended Practice were used for wrought aluminum and wrought aluminum alloys in ASTM specifications prior to 1960, but now only designations conforming to the American Standard Alloy Designation System for Wrought Aluminum (ASA No.: H35.1—1957) are used. The former ASTM designations and the corresponding ASA designations are as follows:

Former ASTM Designation	Corresponding ASA Designation	Former ASTM Designation	Corresponding ASA Designation
990A	1100	GM51A	5456
996A	1060	GR20A	5052
CB60A	2011	GR20B	5652
CG30A	2117	GR40A	5154
CG42A	2024	GR40B	5254
CM41A	2017	GS10A	6063
CN42C	2018	GS10B	6101
CN42D	2218	GS11A	6061
CS41A	2014	GS11B	6053
CS41C	2025	GS11C	6062
G1A	5050	M1A	3003
G1B	5005	MG11A	3004
GM31A	5454	SG11A	6151
GM40A	5086	SG11B	6066
GM41A	5083	SG121A	4032
GM50A	5056	ZG62A	7075

Recommended Practice for Temper Designation of Light Metals and Alloys, Cast and Wrought (B 296 - 56):

Title.—Change to read: "Recommended Practice for Temper Designation of Aluminum and Magnesium Alloys, Cast and Wrought."

Table I.—Change the definitions as shown in the accompanying Table L.[2]

Table II.—Change the definition for −T temper to read: "Thermally treated to produce stable tempers other than −F, −O, or −H."

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Specification for Aluminum-Base Alloy Permanent Mold Castings (**B 108 - 59 T**).

APPENDIX II

DESIGNATIONS FOR METALS AND ALLOYS ASSIGNED IN CONFORMANCE WITH RECOMMENDED PRACTICE FOR CODIFICATION OF LIGHT METALS AND ALLOYS (B 275)

Compiled by Subcommittee VII on Codification of Light Alloys

Designations for metals and alloys assigned in conformance with the Recommended Practice for Codification of Light Metals and Alloys, Cast and Wrought (B 275), and the ASTM Specifications in which they are used, are shown in Tables I, II, and III.

TABLE I.—ALUMINUM BASE.

Designation	ASTM Specification						
	B 26 – 60 T	B 37 – 60 T	B 85 – 60 B	B 108 – 59 T	B 179 – 60	B 285 – 57 T	B 327 – 60 T
850A	. .	*	. .	. .	. .	. .	. .
900A	. .	*	. .	. .	. .	. .	. .
920A	. .	*	. .	. .	. .	. .	. .
950A	. .	*	. .	. .	. .	. .	. .
980A	. .	*	. .	. .	. .	. .	. .
995A	. .	. .	. .	. .	*	. .	. .
C4A	*	. .	. .	. .	*	*	. .
CG100A	*	. .	. .	*	*	. .	. .
CG181A	. .	. .	. .	. .	. .	. .	*
CN42A	*	. .	. .	*	*	*	. .
CS42A	[a]	. .	. .	*	*	. .	. .
CS43A	*	. .	. .	. .	*	. .	. .
CS66A	. .	. .	. .	*	*	. .	. .
CS72A	*	. .	. .	*	*	. .	. .
CS104A	. .	. .	. .	*	*	. .	. .
G1C	. .	. .	. .	. .	. .	. .	*
G4A	*	. .	. .	. .	*	. .	. .
G8A	. .	. .	*	. .	*	. .	. .
G10A	*	. .	. .	. .	*	. .	. .
GM70B	*	. .	. .	*	*	. .	. .
GS42A	*	. .	. .	*	*	. .	. .
GZ42A	. .	. .	. .	*	*	. .	. .
S5A	*	. .	. .	*	*	. .	. .
S5B	*	. .	. .	*	*	*	. .
S5C	. .	. .	*	. .	*	. .	. .
S12A	. .	. .	*	. .	*	. .	. .
S12B	. .	. .	*	. .	*	. .	. .
SC51A	*	. .	. .	*	*	*	. .
SC51B	. .	. .	. .	*	*	. .	. .
SC64A	. .	. .	. .	. .	[a]	. .	. .
SC64C	. .	. .	. .	. .	*	. .	. .
SC64D	*	. .	. .	*	*	. .	. .
SC82A	*	. .	. .	. .	*	. .	. .
SC84A	. .	. .	*	. .	*	. .	. .
SC84B	. .	. .	*	. .	*	. .	. .
SC103A	. .	. .	. .	*	*	. .	. .
SC114A	. .	. .	*	. .	*	. .	. .
SC122A	. .	. .	. .	*	*	. .	. .
SG70A	*	. .	. .	*	*	*	. .
SG70B	. .	. .	. .	*	*	. .	. .
SG100A	. .	. .	*	. .	*	. .	. .
SG100B	. .	. .	*	. .	*	. .	. .
SN122A	. .	. .	. .	*	*	. .	. .
ZC60A	. .	. .	. .	*	*	. .	. .
ZC81A	*	. .	. .	. .	*	. .	. .
ZC81B	. .	. .	. .	*	*	. .	. .
ZG32A	*	. .	. .	*	*	. .	. .
ZG42A	*	. .	. .	*	*	. .	. .
ZG61A	*	. .	. .	. .	*	*	. .
ZG61B	*	. .	. .	. .	*	. .	. .
ZG71A	. .	. .	. .	. .	. .	. .	*

[a] Last appeared in 1958 issue.

TABLE II.—MAGNESIUM BASE.

Designation	ASTM Specification								
	B 80 – 59	B 90 – 60	B 91 – 60	B 92 – 60	B 93 – 60	B 94 – 58	B 107 – 60	B 199 – 59 T	B 217 – 60
9980A	..	..	..	*	..	..	..	..	..
9990A	..	..	..	*	..	..	..	..	..
9995A	..	..	..	*	..	..	..	..	..
[a]A3A	..	..	..	..	..	..	..	..	..
AM80A	..	..	..	..	b	..	..	..	..
AM100A	*	..	..	..	*	..	..	*	..
AM100B	..	..	..	..	b	..	..	..	..
AZ31A	..	b	..	..	..	..	..	..	..
AZ31B	..	*	*	..	..	..	*	..	*
AZ31C	..	*	..	..	..	..	*	..	*
AZ61A	..	..	*	..	..	..	*	..	*
AZ63A	*	..	..	..	*	..	..	..	..
AZ80A	..	..	*	..	..	..	*	..	..
AZ81A	*	..	..	..	*	..	..	..	..
AZ91A	..	..	..	..	*	*	..	..	..
AZ91B	..	..	..	..	*	*	..	..	..
AZ91C	*	..	..	..	*	..	..	*	..
AZ92A	*	..	..	..	*	..	..	*	..
EK30A	*	..	..	..	..	..	..	..	..
EK41A	*	..	..	..	..	..	..	*	..
EZ33A	*	..	..	..	..	..	..	*	..
HK31A	*	*	..	..	..	..	..	..	..
HM21A	..	*	..	..	..	..	..	..	..
[a]HM31A	..	..	..	..	..	..	..	..	..
HZ32A	*	..	..	..	..	..	..	..	..
K1A	*	..	..	..	..	..	..	..	..
M1A	..	..	..	..	..	..	*	..	*
M1B	..	..	..	..	b	..	..	..	..
TA54A	..	..	*	..	..	..	..	..	..
[c]QE22A	..	..	..	..	..	..	..	..	..
ZE10A	..	*	..	..	..	..	..	..	..
ZE41A	*	..	..	..	..	..	..	..	..
ZH62A	*	..	..	..	..	..	..	..	..
[a]ZK21A	..	..	..	..	..	..	..	..	..
ZK51A	*	..	..	..	..	..	..	..	..
ZK60A	..	..	*	..	..	..	*	..	*
[a]ZK60B	..	..	..	..	..	..	..	..	..
ZK61A	*	..	..	..	..	..	..	..	..

[a] Designations reserved for Dow Metal Products Co.

[b] Last appeared in 1957 issue.

[c] Designation reserved for Magnesium Electron, Inc.

TABLE III.—OTHERS.

Designation	ASTM Specification			
	B 176 – 57	B 102 – 52	B 86 – 57 T	B 240 – 57 T
Copper base				
Z30A	*	..	..	..
ZS331A	*	..	..	..
ZS144A	*	..	..	..
Lead base				
YT155A	..	*	..	..
Y10A	..	*	..	..
Tin base				
CY44A	..	*	..	..
PY1815A	..	*	..	..
YC135A	..	*	..	..
Zinc base				
AC41A	..	..	*	*
AG40A	..	..	*	*

APPENDIX III

ATMOSPHERIC EXPOSURE OF WROUGHT ALUMINUM AND MAGNESIUM ALLOYS

By L. H. Adam[1] and M. Dougherty[1]

ASTM Committee B-7 on Light Metals and Alloys is presently accumulating data on aluminum and magnesium alloys that have been exposed at five test sites throughout the country. These ASTM exposure sites, representing rural, industrial, and marine atmospheres, are located at State College, Pa.; New York, N. Y.; Kure Beach, N. C. (East Coast); Point Reyes, Calif. (West Coast); and Freeport, Tex. (Gulf Coast). The program, as outlined in a report submitted to the Society in 1953,[2] includes wrought material and castings which are to be tested for loss of strength and ductility after exposure periods of six months, one, three, five, and ten years. Atmospheric exposure data on the sand and permanent mold castings were reported to the Society in 1954,[3] 1958[4] and 1960,[5] while information on the wrought material was presented in 1955[6] and 1959.[7]

The data submitted in this report pertain to the wrought aluminum and magnesium alloys of sheets (0.064 in.), plates and extruded bars (0.25 in.), plus welded and riveted samples tested after a 5-yr exposure period. Evaluations for the sheets, plates, and extruded bars are based on the average yield strength, tensile strength and per cent elongation determined by testing four standard specimens[8] representing each alloy after exposure at the various test sites. Values for the aluminum riveted samples are based on the average results of six specimens, while the magnesium riveted values represent the average of three specimens per alloy. Tensile strength values are recorded for all fabricated samples with the exception of the spot-welded material. In this instance, only breaking loads are determined. Results are compared with those obtained on similar material which has been retained under control conditions for a 5-yr period.

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[1] Materials Engineer and Physical Science Technician, respectively, U.S. Department of the Army, Ordnance Department, Frankford Arsenal, Philadelphia, Pa.

[2] L. H. Adam, "Atmospheric Exposure of Light Metals," Appendix to Report of Committee B-7 on Light Metals and Alloys, Cast and Wrought, *Proceedings*, Am. Soc. Testing Mats., Vol. 53, p. 227 (1953).

[3] L. H. Adam and M. Dougherty, "Atmospheric Exposure of Aluminum and Magnesium Sand and Permanent Mold Castings," Appendix to Report of Committee B-7 on Light Metals and Alloys, Cast and Wrought, *Proceedings*, Am. Soc. Testing Mats., Vol. 54, p. 270 (1954).

[4] L. H. Adam and M. Dougherty, "Atmospheric Exposure of Aluminum and Magnesium Sand and Permanent Mold Castings," Appendix to Report of Committee B-7 on Light Metals and Alloys, Cast and Wrought, *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 194 (1958).

[5] L. H. Adam and M. Dougherty, "Atmospheric Exposure of Aluminum and Magnesium Sand and Permanent Mold Castings," Appendix to Report of Committee B-7 on Light Metals and Alloys, Cast and Wrought, *Proceedings*, Am. Soc. Testing Mats., Vol. 60, p. 186 (1960).

[6] L. H. Adam, "Atmospheric Exposure of Wrought Aluminum and Magnesium Alloys," Appendix to Report of Committee B-7 on Light Metals and Alloys, Cast and Wrought, *Proceedings*, Am. Soc. Testing Mats. Vol. 55, p. 284 (1955).

[7] L. H. Adam and M. Dougherty, "Atmospheric Exposure of Wrought Aluminum and Magnesium Alloys," Appendix to Report of Committee B-7 on Light Metals and Alloys, Cast and Wrought, *Proceedings*, Am. Soc. Testing Mats. Vol. 59, p. 289 (1959).

[8] Method of Tension Testing of Metallic Materials (E 8), 1958 Book of ASTM Standards, Part 3.

The actual mechanical properties for each alloy are shown in Tables I through XXXIV with the applicable ASTM specifications. Figures 1 to 5 are bar graphs illustrating the average values for each alloy at the five test sites as compared with the average control values for a 5-yr period.

Although observations based on respective control values show no significant difference between the percentage of change after a 5-yr exposure period and that noted after 3-yr exposure, the values recorded after 5-yr exposure generally are lower than those obtained after 3-yr exposure.

The greatest loss of strength and ductility relative to the aluminum alloys is noted in the unclad aluminum-copper sheet, CG42A-T3 (2024), exposed in the marine atmosphere at Point Reyes, Calif. Tensile strength values for four specimens exposed at this particular site for five years averaged 56,900 psi, having an average elongation of 5.3 per cent, while specimens of the same alloy exposed to the rural atmosphere at State College, Pa., averaged 70,300 psi and 18.3 per cent, respectively.

With regard to the magnesium alloys, results indicate that, after 5-yr exposure, the industrial atmosphere at New York City had the greatest deleterious effect on the strength of alloy MlA-O (0.064 in. sheet) when tested as a fabricated and standard specimen.

Through the continuing cooperation of the various laboratories participating in the testing of this material, it has been possible to present exposure data on aluminum and magnesium alloys covering a 5-yr period. The program, as proposed, eventually will cover a ten-year exposure period. This type of information should be of interest to those employed in evaluating the durability of light metals.

3003

TABLE I. - DATA ON ALUMINUM ALLOY NO. M1A-H14 (0.064-IN. SHEET, PANEL).

ASTM Specification Requirements[a]	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
17,000 psi, min.	21,300	21,300	20,200	20,200	21,700	22,200
	21,700	21,400	20,000	20,200	21,200	21,900
	21,700	21,500	19,800	20,500	21,100	22,000
	21,500	21,400	19,900	20,400	20,500	22,000
Average.	21,600	21,400	20,000	20,300	21,100	22,000
TENSILE STRENGTH, PSI						
20,000 psi, min.	22,700	22,600	21,000	21,500	22,800	23,000
26,000 psi, max.	22,700	22,500	20,800	21,700	22,400	22,800
	22,800	22,700	20,900	21,900	22,400	22,800
	22,600	22,700	20,800	21,600	22,200	22,800
Average.	22,700	22,600	20,900	21,700	22,500	22,900
ELONGATION IN 2 IN., PER CENT						
5.0 per cent, min.	8.0	8.0	7.0	7.0	6.0	8.0
	8.0	8.0	7.0	7.0	6.0	7.0
	8.0	8.0	7.0	7.0	6.0	7.0
	7.0	8.0	7.0	7.0	6.0	7.0
Average.	7.8	8.0	7.0	7.0	6.0	7.3

[a] Tentative Specification for Aluminum-Alloy Sheet and Plate (B 209-58T) 1958 Book of ASTM Standards, Part 2

5052

TABLE II. - DATA ON ALUMINUM ALLOY NO. GR20A-H34 (0.064 IN. SHEET, PANEL).

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
26,000 psi, min.	27,700	27,800	26,700	27,100	29,800	28,100
	27,700	27,700	26,500	27,200	28,300	27,600
	27,700	27,800	26,600	27,400	28,000	28,000
	27,800	27,400	26,400	27,600	28,400	27,700
Average.	27,700	27,700	26,600	27,300	28,600	27,900
TENSILE STRENGTH, PSI						
34,000 psi, min.	35,000	34,800	33,100[a]	33,600[a]	35,800	34,700
41,000 psi, max.	34,800	34,500	32,900[a]	33,800[a]	34,800	34,600
	34,900	34,800	33,200[a]	34,000	34,500	35,000
	35,000	34,700	33,000[a]	33,900[a]	34,700	34,800
Average.	34,900	34,700	33,100[a]	33,800[a]	35,000	34,800
ELONGATION IN 2 IN., PER CENT						
6.0 per cent, min.	11.0	10.0	10.0	10.0	8.0	10.0
	10.0	11.0	8.0	10.0	9.0	10.0
	10.0	10.0	10.0	10.0	9.0	9.0
	10.0	10.0	10.0	10.0	9.0	10.0
Average.	10.3	10.3	9.5	10.0	8.8	9.8

[a] Low

6061

TABLE III. - DATA ON ALUMINUM ALLOY NO. GS11A-T6 (0.064 IN. SHEET, PANEL).

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
35,000 psi, min.	40,400	41,000	39,400	39,600	41,600	42,300
	40,500	40,900	38,400	41,600	40,600	42,700
	40,900	41,000	38,400	40,600	40,700	42,700
	40,500	40,700	38,800	41,000	40,900	42,500
Average.	40,600	40,900	38,800	40,700	41,000	42,600
TENSILE STRENGTH, PSI						
42,000 psi, min.	46,900	45,600	43,500	46,200	45,100	46,400
	47,000	45,700	43,100	46,300	44,800	46,300
	46,900	45,500	42,800	46,300	44,800	46,500
	46,800	45,500	43,200	46,400	44,500	46,300
Average.	46,900	45,600	43,200	46,300	44,800	46,400
ELONGATION IN 2 IN., PER CENT						
10.0 per cent min.	14.0	12.0	9.0[a]	11.0	10.0	11.0
	14.0	12.0	10.0	11.0	10.0	11.0
	14.0	12.0	10.0	11.0	9.0[a]	10.0
	13.0	12.0	10.0	11.0	9.0[a]	11.0
Average.	13.8	12.0	9.8[a]	11.0	9.5[a]	10.8

[a] Low

2024

TABLE IV. - DATA ON ALUMINUM ALLOY NO. CG42A-T3 (0.064 IN. SHEET, PANEL).

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
42,000 psi, min.	49,500	49,500	44,500	45,900	44,200	46,400
	49,800	48,900	43,500	46,400	43,800	46,500
	49,800	49,200	45,200	46,600	42,800	46,800
	49,800	49,500	45,300	46,700	43,300	46,400
Average.	49,700	49,300	44,600	46,400	43,500	46,500
TENSILE STRENGTH, PSI						
64,000 psi, min.	70,500	70,200	63,600[a]	67,100	60,100[a]	64,500
	70,300	70,400	62,500[a]	66,100	54,600[a]	64,000
	70,500	70,400	62,700[a]	66,600	56,400[a]	64,900
	70,600	70,300	62,900[a]	66,000	56,400[a]	64,400
Average.	70,500	70,300	62,900	66,500	56,900[a]	64,500
ELONGATION IN 2 IN., PER CENT						
17.0 per cent, min.	20.0	18.0	13.0[a]	14.0[a]	5.0[a]	12.0[a]
	18.0	18.0	13.0[a]	14.0[a]	3.0[a]	12.0[a]
	18.0	18.5	14.0[a]	14.0[a]	7.0[a]	15.0[a]
	20.0	18.5	13.0[a]	14.0[a]	6.0[a]	12.0[a]
Average.	19.0	18.3	13.3[a]	14.0[a]	5.3[a]	12.8[a]

[a] Low

7075

TABLE V. - DATA ON ALUMINUM ALLOY NO. ZG62A-T6 (0.064 IN. SHEET, PANEL).

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
		YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI				
66,000 psi, min.	72,400	71,800	70,300	---	69,100	71,800
	71,500	71,500	69,900	71,600	66,600	71,600
	71,700	71,500	69,900	72,900	66,400	71,600
	71,400	71,800	70,800	72,700	68,300	72,400
Average. . . .	71,800	71,700	70,200	72,400	67,600	71,900
		TENSILE STRENGTH, PSI				
77,000 psi, min.	81,600	81,500	76,500[a]	---	78,300	79,800
	81,800	81,800	76,000[a]	78,400	77,400	79,100
	81,500	81,600	75,700[a]	78,500	78,300	79,100
	81,300	81,500	76,000[a]	79,300	78,500	79,700
Average. . . .	81,600	81,600	76,100[a]	78,700	78,100	79,400
		ELONGATION IN 2 IN., PER CENT				
8.0 per cent, min.	10.0	10.0	9.0	---	9.0	9.0
	10.0	10.0	9.0	9.0	7.0[a]	8.0
	9.0	10.0	9.0	9.0	5.0[a]	9.0
	9.0	10.0	9.0	9.0	7.0[a]	9.0
Average. . . .	9.5	10.0	9.0	9.0	7.0[a]	8.8

3004

TABLE VI. - DATA ON ALUMINUM ALLOY NO. MG11A-H34 (0.064 IN. SHEET, PANEL).

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
		YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI				
25,000 psi, min.	31,500	31,700	33,800	32,500	29,900	30,600
	31,500	31,400	33,400	31,800	30,100	30,100
	31,300	31,700	33,300	31,900	29,800	30,800
	30,700	30,800	33,300	32,000	30,400	31,800
Average. . . .	31,300	31,400	33,500	32,100	30,100	30,800
		TENSILE STRENGTH, PSI				
32,000 psi, min.	36,600	36,700	35,000	35,800	36,000	36,300
38,000 psi, min.	36,500	36,100	34,700	35,600	36,500	36,300
	36,400	36,600	34,300	35,800	36,000	36,200
	36,000	36,000	34,300	35,800	36,400	36,200
Average. . . .	36,400	36,400	34,600	35,800	36,200	36,300
		ELONGATION IN 2 IN., PER CENT				
4.0 per cent, min.	8.0	8.0	8.0	8.0	8.0	9.0
	8.0	8.0	7.0	9.0	8.0	8.0
	8.0	8.0	7.0	8.0	7.0	9.0
	8.0	8.0	8.0	8.0	8.0	8.0
Average. . . .	8.0	8.0	7.5	8.3	7.8	8.5

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[a] Low

5050

TABLE VII. - DATA ON ALUMINUM ALLOY NO. G1A-H34 (0.064 IN. SHEET, PANEL).

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
20,000 psi, min.	23,900	23,800	22,800	23,600	25,700	25,000
	23,700	23,800	22,100	23,500	24,800	25,000
	23,500	23,500	22,500	23,400	24,200	24,700
	23,700	23,800	---	23,600	24,500	25,400
Average.	23,700	23,700	22,500	23,500	24,800	25,000
TENSILE STRENGTH, PSI						
25,000 psi, min.	27,800	27,500	25,600	27,300	27,800	27,800
31,000 psi, max.	27,600	27,400	25,500	27,600	27,700	27,500
	27,500	27,400	25,500	27,400	27,300	27,500
	27,500	27,500	25,700	27,100	27,600	28,100
Average.	27,600	27,500	25,600	27,400	27,600	27,700
ELONGATION IN 2 IN., PER CENT						
5.0 per cent, min.	7.0	7.0	7.0	8.0	7.0	7.0
	7.0	7.0	7.0	8.0	7.0	8.0
	8.0	8.0	7.0	8.0	7.0	8.0
	8.0	8.0	7.0	8.0	7.0	8.0
Average.	7.5	7.5	7.0	8.0	7.0	7.8

2014

TABLE VIII. - DATA ON ALUMINUM ALLOY NO. CS41A-T6 (0.064 IN. CLAD SHEET, PANEL).

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
57,000 psi, min.	60,600	59,100	57,800	60,600	---	60,300
	60,500	58,500	58,000	60,900	60,900	59,900
	60,800	59,700	58,700	59,500	59,100	61,600
	61,000	60,200	57,600	59,500	61,700	59,100
Average.	60,700	59,400	58,000	60,100	60,600	60,200
TENSILE STRENGTH, PSI						
64,000 psi, min.	68,000	66,300	65,200	65,800	62,700[a]	66,400
	68,100	66,500	65,500	65,300	65,000	65,900
	67,800	67,000	64,500	65,200	65,900	66,000
	67,900	67,200	65,000	65,100	66,700	65,800
Average.	68,000	66,800	65,100	65,400	65,100	66,000
ELONGATION IN 2 IN., PER CENT						
8.0 per cent, min.	8.5	8.0	7.0	8.0	8.0	8.0
	9.0	8.0	8.0	9.0	8.0	9.0
	10.0	8.0	8.0	9.0	8.0	8.0
	10.0	8.0	8.0	9.0	8.0	9.0
Average.	9.4	8.0	7.8	8.8	8.0	8.5

[a] Low

2024

TABLE IX. - DATA ON ALUMINUM ALLOY NO. CG42A-T3 (0.064 IN. CLAD SHEET, PANEL).

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
40,000 psi, min.	44,600	44,900	43,300	46,500	45,400	46,200
	46,000	44,800	42,800	44,700	47,800	46,100
	44,600	44,700	44,400	45,000	46,700	46,900
	44,300	45,700	42,800	43,400	46,800	47,400
Average.	44,900	45,000	43,300	44,900	46,800	46,700
TENSILE STRENGTH, PSI						
62,000 psi, min.	67,200	67,100	65,600	66,700	64,600	66,700
	66,800	67,600	63,800	66,900	67,300	66,000
	66,300	67,600	65,400	67,000	66,100	66,500
	66,900	67,400	65,500	66,300	66,900	67,400
Average.	66,800	67,400	65,100	66,700	66,200	66,700
ELONGATION IN 2 IN., PER CENT						
15.0 per cent, min.	20.0	17.0	18.0	19.0	19.0	19.0
	21.0	20.0	18.0	19.0	19.0	19.0
	21.0	21.0	19.0	18.0	20.0	19.0
	19.0	19.0	19.0	20.0	20.0	19.0
Average.	20.3	19.3	18.5	19.0	19.5	19.0

7075

TABLE X. - DATA ON ALUMINUM ALLOY NO. ZG62A-T6 (0.064 IN. CLAD SHEET, PANEL).

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
62,000 psi, min.	68,000	64,600	66,900	73,600	68,100	69,800
	68,800	66,800	65,900	71,100	67,100	69,300
	69,200	66,900	65,600	71,000	68,300	71,100
	68,900	69,100	67,700	71,500	68,000	70,600
Average.	68,700	66,900	66,500	71,800	67,900	70,200
TENSILE STRENGTH, PSI						
72,000 psi, min.	79,300	76,900	76,500	82,000	78,200	78,800
	79,700	78,200	76,100	78,100	76,300	78,800
	80,100	79,300	75,000	77,700	77,800	79,600
	79,100	79,300	75,200	77,900	77,000	79,200
Average.	79,600	78,400	75,700	78,900	77,300	79,100
ELONGATION IN 2 IN., PER CENT						
8.0 per cent, min.	10.0	10.5	12.0	11.0	10.0	9.0
	11.0	11.0	12.0	10.0	10.0	10.0
	10.0	11.0	11.0	11.0	10.0	10.0
	10.0	10.5	10.0	10.0	10.0	10.0
Average.	10.3	10.8	11.3	10.5	10.0	9.8

6061

TABLE XI. - DATA ON ALUMINUM ALLOY NO. GS11A-T6 (0.25 IN. PLATE, PANEL).

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
35,000 psi, min.	42,300	41,400	43,400	42,800	42,400	40,000
	42,200	41,500	45,000	42,000	42,000	39,800
	42,000	41,500	45,800	42,200	41,700	39,500
	42,400	40,900	44,900	42,700	41,700	39,100
Average.	42,200	41,300	44,800	42,400	42,000	39,600
TENSILE STRENGTH, PSI						
42,000 psi, min.	46,700	46,300	48,300	46,300	47,200	46,500
	46,300	46,000	48,300	46,300	47,300	46,600
	46,300	46,200	48,300	46,100	47,300	46,200
	46,800	45,800	48,300	46,200	47,100	46,400
Average.	46,500	46,100	48,300	46,200	47,200	46,400
ELONGATION IN 2 IN., PER CENT						
10.0 per cent, min.	17.0	17.0	15.0	15.0	15.0	16.0
	16.0	17.0	14.0	16.0	16.0	16.0
	17.0	17.0	14.0	16.0	15.0	17.0
	17.0	16.0	14.0	16.0	16.0	17.0
Average.	16.8	16.8	14.8	15.8	15.5	16.5

2014

TABLE XII. - DATA ON ALUMINUM ALLOY NO. CS41A-T4 (0.25 IN. PLATE, PANEL).

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
No Requirement	49,200	50,600	49,800	51,000	41,100	41,300
	49,000	48,300	47,300	45,000	41,700	41,700
	47,900	48,000	47,300	51,400	40,900	42,100
	48,300	51,000	46,600	46,700	41,100	41,800
Average.	48,600	49,500	47,800	48,500	41,200	41,700
TENSILE STRENGTH, PSI						
No Requirement	66,300	66,100	65,400	66,200	62,400	66,600
	66,200	66,200	66,200	65,500	63,600	66,000
	66,100	66,300	64,600	65,700	61,400	65,500
	65,800	66,300	65,900	66,400	61,600	63,500
Average.	66,100	66,200	65,500	66,000	62,300	65,400
ELONGATION IN 2 IN., PER CENT						
No Requirement	17.0	18.0	15.0	15.0	11.0	16.0
	19.0	18.0	16.0	13.0	11.0	16.0
	18.0	17.0	16.0	13.0	10.0	12.0
	18.0	17.0	16.0	14.0	10.0	12.0
Average.	18.0	17.5	15.8	13.8	10.5	14.0

2014

TABLE XIII. - DATA ON ALUMINUM ALLOY NO. CS41A-T6 (0.064 IN. PLATE, PANEL).

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
No Requirement	66,800	67,800	66,100	67,200	60,100	63,200
	67,400	67,200	66,400	66,400	58,400	63,400
	67,700	68,300	66,000	67,500	58,500	64,500
	67,500	68,000	64,600	66,500	59,000	63,800
Average.	67,400	67,800	65,800	66,900	59,000	63,700
TENSILE STRENGTH, PSI						
No Requirement	72,100	71,900	71,300	70,700	67,000	71,200
	71,800	71,600	72,000	70,600	65,400	71,400
	71,500	72,100	70,700	70,700	64,900	71,700
	71,500	71,200	70,500	71,200	65,800	71,500
Average.	71,700	71,700	71,100	70,800	65,800	71,500
ELONGATION IN 2 IN., PER CENT						
No Requirement	9.0	9.0	6.0	7.0	6.0	7.0
	9.0	9.0	6.0	6.0	7.0	7.0
	9.0	9.0	6.0	7.0	5.0	8.0
	9.0	8.5	6.0	7.0	5.0	7.0
Average.	9.0	8.9	6.0	6.8	5.8	7.3

2014

TABLE XIV. - DATA ON ALUMINUM ALLOY NO. CS41A-T6 (0.25 IN. CLAD PLATE, PANEL).

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
YIELD STRENGTH 0.2 PER CENT OFFSET, PSI						
57,000 psi, min.	63,000	62,900	62,300	63,400	58,900	58,900
	63,000	63,500	60,700	63,500	58,700	59,200
	62,300	64,200	59,500	61,900	59,300	58,600
	62,200	63,500	61,400	63,000	59,600	58,500
Average.	62,600	63,500	61,000	63,000	59,100	58,800
TENSILE STRENGTH, PSI						
64,000 psi, min.	67,200	67,100	67,000	66,500	67,000	66,900
	66,900	67,900	66,600	66,900	66,300	66,600
	66,700	67,600	65,700	67,300	66,600	66,700
	67,200	67,700	66,600	67,400	66,800	66,600
Average.	67,000	67,600	66,500	67,000	66,700	66,700
ELONGATION IN 2 IN., PER CENT						
8.0 per cent, min.	10.0	9.0	8.0	8.0	10.0	9.0
	11.0	9.0	9.0	8.0	9.0	11.0
	10.0	9.0	9.0	9.0	9.0	9.0
	10.0	9.0	9.0	9.0	9.0	10.0
Average.	10.3	9.0	8.8	8.5	9.3	9.8

2024

TABLE XV. - DATA ON ALUMINUM ALLOY NO. CG42A-T4 (0.25 IN. CLAD PLATE, PANEL).

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
		YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI				
40,000 psi, min.	47,800	48,000	51,100	52,600	45,600	43,300
	48,200	47,900	52,300	54,600	45,900	44,000
	47,800	48,400	50,600	52,400	45,200	43,500
	48,600	47,700	52,700	51,400	45,300	44,000
Average.	48,100	48,000	51,700	52,800	45,500	43,700
		TENSILE STRENGTH, PSI				
62,000 psi, min.	65,800	65,000	64,700	67,000	66,200	66,200
	66,500	65,200	65,100	67,500	66,700	67,100
	66,100	65,800	65,000	67,800	66,200	66,400
	66,800	65,500	65,200	68,300	63,400	66,300
Average. . . .	66,300	65,400	65,000	67,700	65,600	66,500
		ELONGATION IN 2 IN., PER CENT				
11.0 per cent, min.	19.0	19.0	16.0	16.0	17.0	18.0
	18.0	17.0	17.0	18.0	17.0	18.0
	19.0	17.0	17.0	18.0	17.0	18.0
	19.0	18.0	16.0	18.0	17.0	19.0
Average.	18.8	17.8	16.5	17.5	17.0	18.3

7075

TABLE XVI. DATA ON ALUMINUM ALLOY NO. ZG62A-T6 (0.25 IN. CLAD PLATE, PANEL)

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
		YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI				
62,000 psi, min.	71,100	73,000	75,000	72,000	67,500	67,300
	71,700	72,200	76,100	72,800	65,700	67,700
	72,600	70,900	75,700	71,700	67,400	68,100
	71,100	72,200	71,700	70,500	67,600	68,500
Average.	71,600	72,100	74,600	71,800	67,100	67,900
		TENSILE STRENGTH, PSI				
72,000 psi, min.	77,000	77,000	77,300	76,900	77,300	76,900
	77,000	77,300	77,100	77,500	77,300	77,200
	77,400	77,300	77,400	76,100	77,300	77,700
	76,700	77,200	77,000	78,000	76,700	77,800
Average.	77,000	77,200	77,200	77,100	77,200	77,400
		ELONGATION IN 2 IN., PER CENT				
3.0 per cent, min.	11.0	9.0	9.0	9.0	8.0	10.0
	10.0	9.0	9.0	9.0	8.0	10.0
	10.0	10.0	9.0	9.0	8.0	10.0
	10.0	10.0	9.0	9.0	8.0	10.0
Average.	10.3	9.5	9.0	9.0	8.0	10.0

6061

TABLE XVII. DATA ON ALUMINUM ALLOY NO. GS11A-T6 (0.25 IN. EXTRUDED BAR, PANEL).

ASTM Specification Requirements[a]	5-YR CONTROL	5 - YEAR EXPOSURE STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
35,000 psi, min.	40,500 40,600 40,700 41,100	40,700 41,000 40,300 40,200	40,700 39,800 39,300 40,000	41,400 41,100 40,200 40,700	40,400 40,500 40,300 41,000	40,300 39,500 39,900 40,300
Average.	40,700	40,600	40,000	40,900	40,600	40,000
TENSILE STRENGTH, PSI						
38,000 psi, min.	43,700 43,600 43,600 43,900	43,600 43,500 43,300 43,300	43,200 42,400 42,100 42,600	43,800 43,400 42,500 43,500	43,700 43,200 43,100 44,000	43,200 43,100 43,500 43,000
Average.	43,700	43,400	42,600	43,300	43,500	43,200
ELONGATION IN 2 IN., PER CENT						
10.0 per cent, min.	16.0 16.0 16.0 16.0	16.0 15.0 15.0 15.0	14.0 14.0 14.0 14.0	15.0 15.0 15.0 15.0	14.0 15.0 15.0 15.0	16.0 16.0 14.0 15.0
Average.	16.0	15.3	14.0	15.0	14.8	15.3

6063

TABLE XVIII. DATA ON ALUMINUM ALLOY NO. GS10A-T5 (0.25 IN. EXTRUDED BAR, PANEL).

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
16,000 psi, min.	25,600 26,000 26,700 26,700	25,600 26,600 25,400 25,400	29,800 29,700 29,800 29,000	27,800 28,200 29,400 29,000	26,500 26,700 26,900 26,800	28,200 28,000 28,000 27,400
Average.	26,300	25,800	29,600	28,600	26,700	27,900
TENSILE STRENGTH, PSI						
22,000 psi, min.	29,700 29,800 30,300 30,300	29,800 29,600 29,600 29,700	32,400 33,000 32,800 32,600	31,700 31,800 32,200 31,800	30,900 30,900 31,400 30,900	32,200 32,600 31,900 32,300
Average.	30,000	29,700	32,700	31,900	31,000	32,300
ELONGATION IN 2 IN., PER CENT						
8.0 per cent, min.	16.0 16.0 16.0 16.0	16.0 17.0 17.0 17.0	14.0 14.0 14.0 14.0	15.0 14.0 14.0 14.0	14.0 15.0 15.0 15.0	15.0 15.0 15.0 16.0
Average.	16.0	16.8	14.0	14.3	14.8	15.3

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[a] Tentative Specifications for Aluminum-Alloy Extruded Bars, Rods, and Shapes (B 221-58T)

1958 Book of ASTM Standards, Part 2.

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TABLE XIX. DATA ON ALUMINUM ALLOY NO. CS41A-T4 (0.25 IN. EXTRUDED BAR, PANEL).

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
		YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI				
35,000 psi, min.	41,700	40,700	40,600	41,200	38,800	41,900
	42,400	42,200	42,800	44,500	40,700	43,100
	44,800	44,200	44,700	43,800	41,400	42,100
	43,900	43,500	44,000	42,200	41,300	38,400
Average	43,200	42,700	43,000	42,900	40,600	41,400
		TENSILE STRENGTH, PSI				
50,000 psi, min.	56,400	55,600	56,000	54,700	54,400	59,200
	57,700	56,900	57,900	57,200	56,200	59,300
	58,000	57,900	59,600	58,000	56,300	59,700
	58,000	57,900	59,100	56,900	56,900	56,100
Average	57,500	57,100	58,200	56,700	56,000	58,600
		ELONGATION IN 2 IN., PER CENT				
12.0 per cent, min.	28.0	27.0	24.0	21.0	20.0	21.0
	24.0	25.0	23.0	21.0	24.0	16.0
	24.0	22.0	22.0	22.0	20.0	22.0
	24.0	25.0	21.0	21.0	21.0	23.0
Average	25.0	24.8	22.5	21.3	21.3	20.5

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TABLE XX. DATA ON ALUMINUM ALLOY NO. CS41A-T6 (0.25 IN. EXTRUDED BAR, PANEL).

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
		YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI				
53,000 psi, min.	61,300	59,700	60,200	58,900	57,300	61,400
	61,300	62,200	62,400	62,300	60,300	60,700
	62,500	61,800	62,900	62,000	61,000	61,300
	63,300	62,700	61,800	63,300	60,000	57,700
Average	62,100	61,600	61,800	61,600	59,700	60,300
		TENSILE STRENGTH, PSI				
60,000 psi, min.	64,300	63,600	63,100	62,400	61,900	65,000
	65,100	65,900	65,400	64,400	64,000	64,800
	65,800	64,600	65,600	65,000	64,400	64,900
	66,000	65,500	65,000	65,300	63,900	62,300
Average	65,300	64,900	64,800	64,300	63,600	64,300
		ELONGATION IN 2 IN., PER CENT				
7.0 per cent, min.	14.0	14.0	10.0	11.0	10.0	10.0
	13.0	15.0	10.0	11.0	9.0	10.0
	11.0	8.0	10.0	11.0	9.0	10.0
	11.0	11.0	12.0	11.0	10.0	12.0
Average	12.3	12.0	10.5	11.0	9.5	10.5

7075

TABLE XXI. DATA ON ALUMINUM ALLOY NO. ZG62A-T6 (0.25 IN. EXTRUDED BAR, PANEL).

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
		YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI				
72,000 psi, min.	—	84,200	—	89,400	84,300	80,900
	85,000	82,800	83,600	85,200	79,600	80,700
	83,600	81,800	81,800	83,900	76,800	82,600
	85,700	84,200	77,000	—	77,400	84,800
Average.	84,800	83,300	80,800	86,200	79,500	82,300
		TENSILE STRENGTH, PSI				
80,000 psi, min.	92,000	88,200	91,100	93,200	89,500	88,000
	89,100	87,400	87,900	89,900	86,200	87,500
	87,600	86,100	86,600	87,900	83,400	89,300
	89,400	86,200	89,200	90,000	86,200	90,000
Average.	89,500	87,500	88,700	90,300	86,300	88,700
		ELONGATION IN 2 IN., PER CENT				
7.0 per cent, min.	12.0	12.0	8.0	8.0	6.0[a]	9.0
	10.0	11.0	7.0	8.0	6.0[a]	9.0
	12.0	10.0	7.0	8.0	6.0[a]	9.0
	9.0	10.0	7.0	8.0	7.0	9.0
Average.	10.8	10.8	7.3	8.0	6.3[a]	9.0

a - Low

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TABLE XXII. DATA ON ALUMINUM ALLOYS NOS. GS11A-T6, CS41A-T6, CG42A-T3, AND ZG62A-T6 (0.064 IN. SHEET, SPOT WELD[a]).

7075

	BREAKING LOAD, LB 5-YR CONTROL	5 - YEAR EXPOSURE STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
		ALLOY NO. GS11A-T6				
	1419	1349	—	—	1724	1810
	1404	1388			1624	1250
	1214	1439			1633	1120
	1241	1260			1432	—
Average.	1320	1359			1603	1393
		ALLOY NO. CS41A-T6 - CLAD				
	1247	1447	—	—	1995	2165
	1590	1535			1713	—
	1580	1342			1913	—
	1539	1316			1992	1740
Average.	1489	1410			1903	1953
		ALLOY NO. CG42A-T3 - CLAD				
	1555	1630	—	—	2000	1920
	1485	1700			1998	1230
	1345	1240			1943	1090
	1740	1415			1633	1300
Average.	1531	1496			1894	1385
		ALLOY NO. ZG62A-T6 - CLAD				
	2310	2535	2035	2754	1900	2381
	2490	2375	1762	2488	1845	2106
	2420	2217	2060	2396	1740	2365
	2415	2240	2150	2204	2130	2110
Average.	2409	2342	2002	2461	1904	2241

[a] Values are shear results for 2 spot welds on a single lap joint.

TABLE XXIII. DATA ON ALUMINUM ALLOYS NOS. $\overset{6061}{\text{GS11A-T6}}$ AND $\overset{6063}{\text{GS10A-T5}}$.

	TENSILE STRENGTH, PSI					
	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
	ALLOY NO. GS11A-T6 (0.25 IN. PLATE, GAS WELD)					
	22,600	21,900	24,500	25,200	26,300[a]	26,900
	27,000	22,400	24,400	25,500	25,300[a]	21,500
	26,800	23,300	24,300	25,800	27,600	25,200
	21,800	23,400	24,400	25,800	24,300[a]	26,100
Average . . .	24,600	22,800	24,400	25,600	25,900	24,900
	ALLOY NO. GS11A-T6 (0.25 IN. PLATE, ARC WELD)					
	25,000	22,900	22,600	28,100	22,900	22,100
	25,800	23,100	22,600	28,400	21,900	22,500
	27,700	22,400	21,300	26,800	21,900	21,800
	25,600	21,700	22,800	25,900	21,600	22,700
Average	26,000	22,500	22,300	27,300	22,100	22,300
	ALLOY NO. GS10A-T5 (0.25 IN. EXTRUDED BAR, ARC WELD)					
	20,900	21,100	20,500	21,200	20,400	20,100
	20,900	21,100	20,800	21,700	21,100	20,400
	21,200	21,300	20,800	21,800	21,100	20,400
	20,800	21,300	20,600	21,700	20,900	20,400
Average . . .	21,000	21,200	20,700	21,600	20,900	20,300

[a] Samples failed through weld.
All other samples failed in base material.

TABLE XXIV. - DATA ON ALUMINUM ALLOYS, NOS. $\overset{6061}{\text{GS11A-T6}}$, $\overset{2024}{\text{CG42A-T3}}$, AND $\underset{7075}{\text{ZG62A-T6}}$ (0.064 IN. SHEET, RIVET[a, b])

	TENSILE STRENGTH, PSI					
	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
	ALLOY NO. GS11A-T6					
	46,000	46,400	46,800	44,900	47,000	45,200
	46,400	46,200	46,400	46,600	48,200	44,500
	46,000	46,200	45,800	46,800	47,000	43,900
	46,400	47,500	46,600	45,800	46,900	45,100
	43,300	46,800	46,800	46,000	46,400	46,200
	45,800	47,500	45,200	45,600	44,800	44,800
Average. . . .	45,700	46,800	46,300	46,000	46,700	45,000
	ALLOY NO. CG42A-T3					
	--	63,100	60,800	59,400	51,100[c]	53,400
	61,000	61,000	61,500	58,800	29,000[c]	54,500
	61,600	63,600	62,600	60,900	44,900[c]	59,900
	61,400	57,800	62,400	60,500	15,400[c]	55,300
	61,000	63,300	63,500	61,800	46,200[c]	57,800
	61,000	63,400	60,900	62,600	22,000[c]	61,100
Average. . . .	61,200	62,000	62,000	60,700	34,800	57,000
	ALLOY NO. ZG62A-T6					
	77,200	73,400	79,900	80,600	73,300	78,400
	77,600	80,200	80,400	80,000	72,900	79,900
	58,400	78,500	79,600	80,000	75,000	78,600
	86,700	84,800	79,300	81,700	78,400	79,000
	75,900	78,500	79,200	80,000	79,600	78,600
	76,700	75,100	79,900	81,700	80,400	78,000
Average. . . .	75,400	78,400	79,700	80,700	76,600	78,800

[a] Net area = Width - diameter of rivet hole x thickness.
[b] All specimens failed in sheet.
[c] Samples badly corroded.

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TABLE XXV. - DATA ON ALUMINUM ALLOYS, NOS. CS41A-T6, CG42A-T3, AND
ZG62A-T6 (0.064 IN. CLAD SHEET, RIVET[a],[b])
7075

TENSILE STRENGTH, PSI

	5-YR CONTROL	5 - YEAR EXPOSURE STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
		ALLOY NO. CS41A-T6				
	58,000 65,700 51,000 65,200 75,500 68,800	62,900 65,800 70,700 62,900 65,800 63,900	67,400 64,200 66,200 64,500 64,800 65,400	67,800 68,100 68,700 68,500 68,500 69,900	67,900 67,700 68,000 67,300 68,100 66,700	67,200 67,500 68,600 67,200 67,000 66,800
Average. . . .	64,000	65,300	65,400	68,600	67,600	67,400
		ALLOY NO. CG42A-T3				
	63,300 60,800 55,600 65,200 62,900 63,900	59,300 63,300 59,600 64,600 62,700 65,600	63,300 62,800 63,100 62,500 62,400 60,900	62,900 61,100 62,200 61,300 61,300 62,100	59,500 61,700 62,900 63,000 61,200 60,500	63,500 61,000 61,800 62,500 61,300 62,500
Average. . . .	62,000	62,500	62,500	61,800	61,500	62,100
		ALLOY NO. ZG62A-T6				
	75,300 75,100 84,500 74,500 65,900 71,400	78,200 79,300 73,300 77,200 75,500 83,200	76,400 74,400 75,000 76,000 75,400 75,800	79,400 77,900 79,300 80,000 79,600 80,200	81,500 79,100 80,600 80,100 81,300 78,500	81,400 80,100 78,200 79,300 81,500 78,800
Average. . . .	74,500	77,800	75,500	79,400	80,200	79,900

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[a] Net area = Width - diameter of rivet hole x thickness.
[b] All specimens failed in sheet.

TABLE XXVI. - DATA ON MAGNESIUM ALLOY NO. M1A-O (0.064 IN. SHEET, PANEL).

ASTM Specification Requirements[a]	5-YR CONTROL	5 - YEAR EXPOSURE STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
			YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI			
No Requirements	15,100 15,600 15,600 15,400	15,400 15,300 15,200 15,000	11,600 11,000 11,700 11,000	14,200 14,200 14,200 14,500	15,300 15,400 15,700 15,300	16,600 16,900 16,300 15,700
Average.	15,400	15,200	11,300	14,300	15,400	16,400
			TENSILE STRENGTH, PSI			
No Requirements	30,900 31,100 31,400 31,000	25,500 24,600 24,500 24,200	22,400 22,300 22,500 22,000	26,200 26,000 25,900 26,400	27,000 26,400 27,300 26,300	28,100 28,100 28,400 28,700
Average.	31,100	24,700	22,300	26,100	26,800	28,300
			ELONGATION IN 2 IN., PER CENT			
No Requirements	18.5 18.5 18.0 18.5	3.5 3.5 3.5 —	8.0 10.0 8.0 9.0	— 5.5 5.0 5.5	5.0 4.0 5.0 4.5	-- -- 6.5 6.0
Average.	18.4	3.5	8.8	5.3	4.6	6.3

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[a] Standard Specification for Magnesium-Base Alloy Sheet and Plate (B 90-58)

1958 Book of ASTM Standards, Part 2.

TABLE XXVII. - DATA ON MAGNESIUM ALLOY NO. AZ31B-H24 (0.064 IN. SHEET, PANEL).

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
29,000 psi, min.	29,300	28,700[a]	26,300[a]	29,000	27,700[a]	29,900
	29,300	28,200[a]	26,700[a]	29,000	27,800[a]	29,700
	29,700	28,100[a]	26,700[a]	29,100	28,100[a]	30,200
	29,300	28,400[a]	26,500[a]	29,300	28,100[a]	29,700
Average. . . .	29,400	28,400[a]	26,600[a]	29,100	27,900[a]	29,900
TENSILE STRENGTH, PSI						
39,000 psi, min.	42,600	38,300[a]	33,900[a]	39,800	39,100	41,000
	40,800	38,700[a]	33,100[a]	40,000	39,200	40,800
	41,200	38,700[a]	34,000[a]	39,600	39,200	41,100
	40,600	37,800[a]	33,700[a]	39,600	39,100	40,900
Average. . . .	41,300	38,400[a]	33,700[a]	39,800	39,200	41,000
ELONGATION IN 2 IN., PER CENT						
4.0 per cent, min.	16.5	6.5	10.5	7.0	7.5	9.5
	16.5	6.5	11.0	7.0	7.5	9.5
	16.5	—	12.5	8.5	9.0	6.5
	17.5	7.5	10.5	7.0	7.0	9.5
Average. . . .	16.8	6,8	11.1	7.4	7.8	8.8

[a] Low

TABLE XXVIII. - DATA ON MAGNESIUM ALLOY NO. AZ31B-H24 (0.25 IN. PLATE, PANEL).

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
29,000 psi, min.	32,300	31,000	28,400[a]	31,300	31,100	31,900
	32,100	30,400	28,700[a]	31,300	31,200	31,800
	31,300	31,500	27,900[a]	31,300	31,200	31,900
	33,000	31,200	28,700[a]	31,300	30,500	31,900
Average.	32,200	31,000	28,400[a]	31,300	31,000	31,900
TENSILE STRENGTH, PSI						
39,000 psi, min.	41,400	40,300	39,200	41,300	40,700	41,500
	41,600	40,500	39,200	41,200	40,600	41,400
	41,200	40,400	39,100	41,100	39,300	41,400
	41,000	40,500	39,100	41,200	40,500	41,500
Average.	41,300	40,400	39,200	41,200	40,300	41,500
ELONGATION IN 2 IN., PER CENT						
4.0 per cent, min.	19.0	10.0	11.5	9.0	9.5	12.5
	19.0	10.0	12.5	10.0	8.5	11.0
	18.0	—	—	9.0	5.5	10.5
	—	10.0	11.5	10.0	8.0	11.0
Average.	18.7	10.0	11.8	9.5	7.8	11.3

[a] Low

TABLE XXIX. - DATA ON MAGNESIUM ALLOY NO. M1A-F (0.25 IN. EXTRUDED BAR).

ASTM Specification Requirements[a]	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
No Requirement	—	22,000	21,000	22,100	21,000	23,400
	—	21,000	21,000	22,600	21,400	22,100
	21,600	20,800	21,400	20,800	21,200	21,200
	21,600	22,700	21,000	21,000	21,200	23,400
Average.	21,600	21,600	21,100	21,600	21,200	22,500
TENSILE STRENGTH, PSI						
32,000 psi, min.	33,700	29,400[b]	30,600[b]	30,700[b]	31,200[b]	31,800[b]
	33,600	28,600[b]	30,400[b]	31,100[b]	30,700[b]	32,000
	32,900	29,800[b]	30,400[b]	31,200[b]	30,900[b]	32,100
	32,800	30,500[b]	29,900[b]	30,500[b]	31,600[b]	33,200
Average.	33,300	29,600[b]	30,300[b]	30,900[b]	31,100[b]	32,200
ELONGATION IN 2 IN., PER CENT						
3.0 per cent, min.	15.0	4.0	—	5.0	7.0	—
	15.0	4.0	5.0	5.0	5.5	6.5
	13.0	—	—	5.0	5.0	—
	10.0	—	4.5	5.0	5.0	—
Average.	13.3	4.0	4.8	5.0	5.6	6.5

TABLE XXX. - DATA ON MAGNESIUM ALLOY NO. AZ31B-F (0.25 IN. EXTRUDED BAR).

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
22,000 psi, min.	24,400	25,500	24,600	25,300	24,700	25,000
	—	25,500	24,100	25,200	24,700	24,700
	24,400	26,000	24,500	25,300	24,600	24,800
	25,000	—	24,700	25,500	24,700	24,700
Average.	24,600	25,700	24,500	25,300	24,700	24,800
TENSILE STRENGTH, PSI						
35,000 psi, min.	38,100	36,700	35,900	37,400	37,500	38,000
	38,600	36,800	35,800	37,400	37,400	38,300
	37,700	36,800	35,800	37,300	37,500	37,900
	38,300	—	35,100	37,400	37,300	38,200
Average.	38,200	36,800	35,700	37,400	37,400	38,100
ELONGATION IN 2 IN., PER CENT						
7.0 per cent, min.	15.5	13.5	12.5	13.5	12.0	16.5
	16.5	13.5	12.0	—	13.5	16.5
	16.5	13.0	12.5	14.0	12.5	17.0
	16.5	—	10.0	12.5	13.0	16.5
Average.	16.3	13.3	11.8	13.3	12.8	16.6

[a] Standard Specification for Magnesium-Base Alloy Bars, Rods and Shapes (B 107-58)
1958 Book of ASTM Standards, Part 2

[b] Low

TABLE XXXI. - DATA ON MAGNESIUM ALLOY NO. AZ80A-T51 (0.25 IN. EXTRUDED BAR).

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
33,000 psi, min.	37,700	38,000	35,400	38,000	36,500	38,100
	36,700	38,800	35,600	38,100	36,700	38,500
	38,300	38,800	34,800	38,600	36,500	39,300
	38,100	38,500	35,600	38,800	36,800	39,400
Average.	37,700	38,500	35,400	38,400	36,600	38,800
TENSILE STRENGTH, PSI						
48,000 psi, min.	53,500	51,100	46,300	51,500	47,700	52,700
	53,700	52,200	46,600	50,600	48,300	53,100
	53,700	50,600	46,600	49,500	47,900	52,300
	53,600	50,300	47,000	49,900	47,600	52,200
Average.	53,600	51,100	46,600	50,400	47,900	52,600
ELONGATION IN 2 IN., PER CENT						
4.0 per cent, min.	10.0	—	—	3.5[a]	4.5	6.5
	11.0	6.5	4.0	3.5[a]	4.0	7.5
	7.5	5.0	4.0	3.5[a]	4.0	6.5
	6.0	5.0	4.0	—	4.0	6.0
Average.	8.6	5.5	4.0	3.5[a]	4.1	6.6

[a] Low

TABLE XXXII. - DATA ON MAGNESIUM ALLOY NO. ZK60A-F (0.25 IN. EXTRUDED BAR).

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
31,000 psi, min.	36,600	34,400	33,100	36,000	35,500	38,200
	36,300	35,300	33,800	37,100	35,400	38,300
	36,400	34,100	33,400	36,400	35,700	37,900
	36,700	35,500	32,600	36,600	35,900	38,100
Average.	36,500	34,800	33,200	36,500	35,600	38,100
TENSILE STRENGTH, PSI						
43,000 psi, min.	48,300	45,600	44,100	46,300	45,800	47,600
	47,600	45,400	44,300	47,600	45,800	47,300
	47,600	45,200	44,200	47,200	45,700	47,700
	48,100	45,500	43,800	47,200	45,700	47,600
Average.	47,900	45,400	44,100	47,100	45,800	47,600
ELONGATION IN 2 IN. PER CENT						
5.0 per cent, min.	9.5	9.0	11.0	10.5	10.0	11.0
	7.5	—	11.0	10.5	10.0	11.5
	10.0	9.5	15.0	10.5	10.0	11.5
	10.5	10.0	12.0	10.5	10.0	10.5
Average.	9.4	9.5	12.3	10.5	10.0	11.1

TABLE XXXIII. - DATA ON MAGNESIUM ALLOYS NOS. M1A-0, AZ31B-H24 (0.064 IN. SHEET, SPOT WELD).

	BREAKING LOAD, LB					
	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
		ALLOY NO. M1A - 0				
	665	555	475	525	605	540
	530	495	425	450	--	445
	485	--	370	410	--	--
	580	440	375	510	525	520
Average. . . .	565	497	411	474	565	500
		ALLOY NO. AZ31B - H24				
	--	610	765	--	830	755
	--	650	625	--	600	600
	530	655	--	710	645	615
	625	720	--	760	740	800
Average.	578	659	695	735	704	690

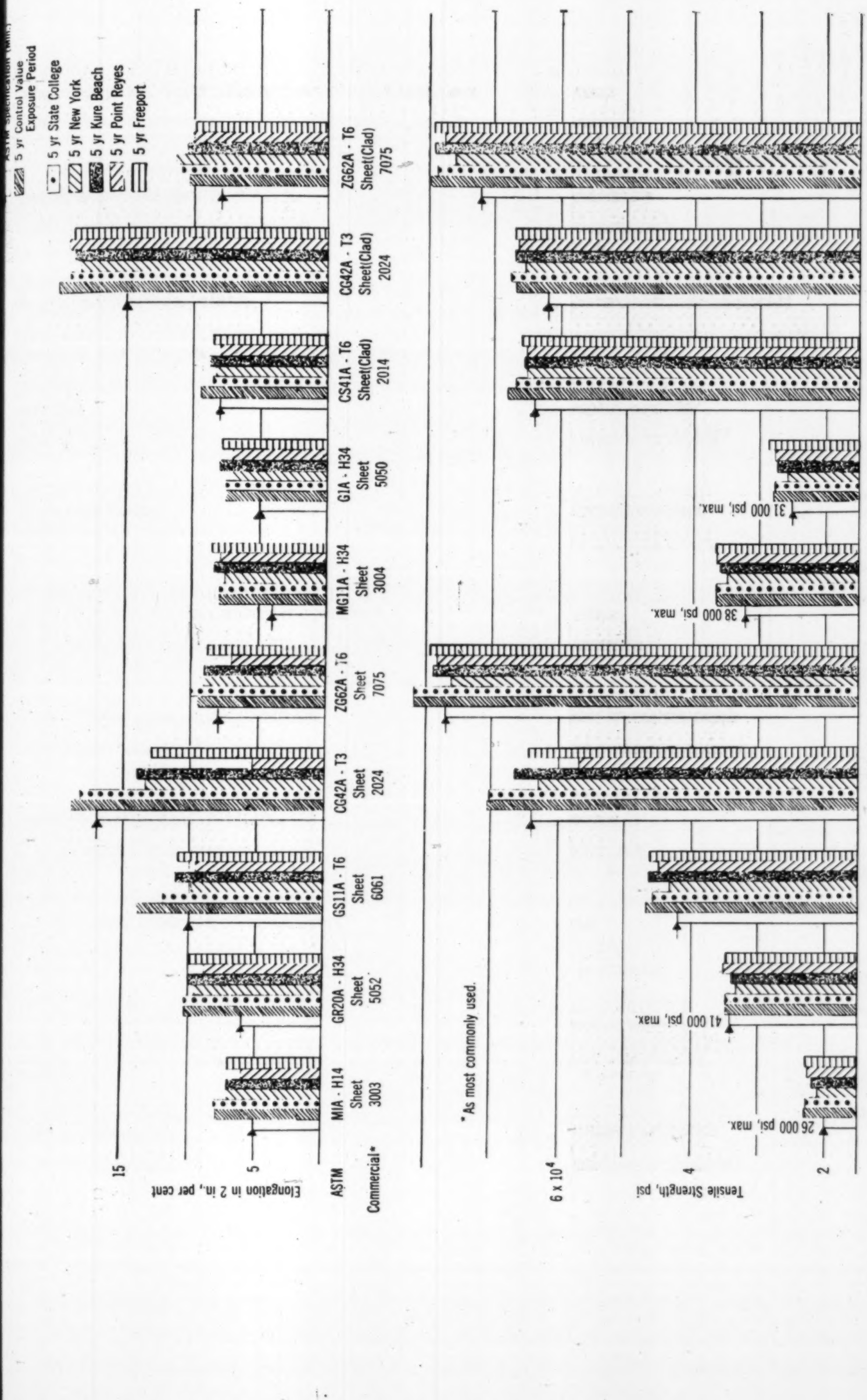
Values are shear results for 2 spot welds on a single lap joint.

TABLE XXXIV. - DATA ON MAGNESIUM ALLOYS,
NOS. M1A-0 and AZ31B-H24 (0.064 IN. SHEET, RIVET[a,b])
NOS. AZ31B-F and ZK60A-F (0.25 IN. EXTRUDED BAR, RIVET[a,b])

	TENSILE STRENGTH, PSI					
	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
		ALLOY NO. M1A - 0				
	27,500	28,800	24,900	30,500	28,300	28,300
	27,600	27,000	28,300	30,600	28,600	29,400
	27,300	28,400	27,500	30,800	28,400	28,100
Average.	27,500	28,100	26,900	30,600	28,500	28,600
		ALLOY NO. AZ31B - H24				
	42,000	46,400	40,800	40,900	31,800	—
	43,500	44,500	40,900	41,100	43,200	
	43,900	46,000	40,200	41,700	22,300	
Average. . . .	43,100	45,600	40,600	41,200	32,400	
		ALLOY NO. AZ31B - F				
	38,100	37,800	35,600	35,700	33,800	40,100
	39,200	37,400	37,700	37,500	38,600	38,500
	37,400	37,800	37,400	36,600	36,600	37,100
Average.	38,200	37,700	36,900	36,600	36,300	38,600
		ALLOY NO. ZK60A - F				
	44,800	—	45,300	42,500	43,000	45,500
	42,900		43,600	45,000	43,100	44,000
	44,800		42,900	45,600	43,800	42,700
Average. . . .	44,200		43,900	44,400	43,300	44,100

[a] Net area = Width - diameter of rivet hole x thickness.

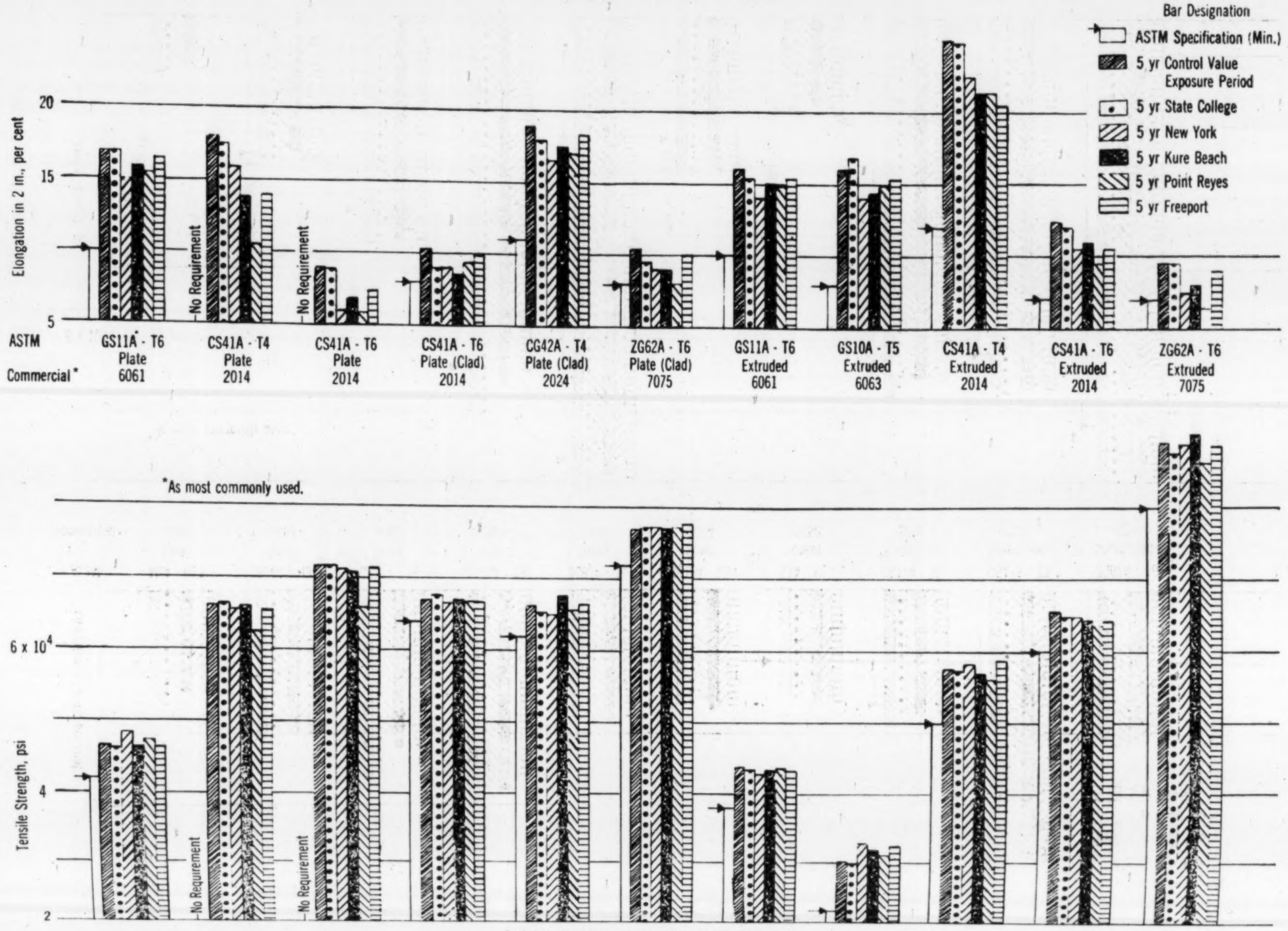
[b] All specimens failed in sheet.



* As most commonly used.

Each bar represents average results of four specimens.

FIG. 1.—Summary of Data, Atmospheric Exposure of Light Metals and Alloys, Aluminum Panels 0.064 in.



*As most commonly used.

Each bar represents average results of four specimens.

FIG. 2.—Summary of Data, Atmospheric Exposure of Light Metals and Alloys, Aluminum Alloys 0.25 in.

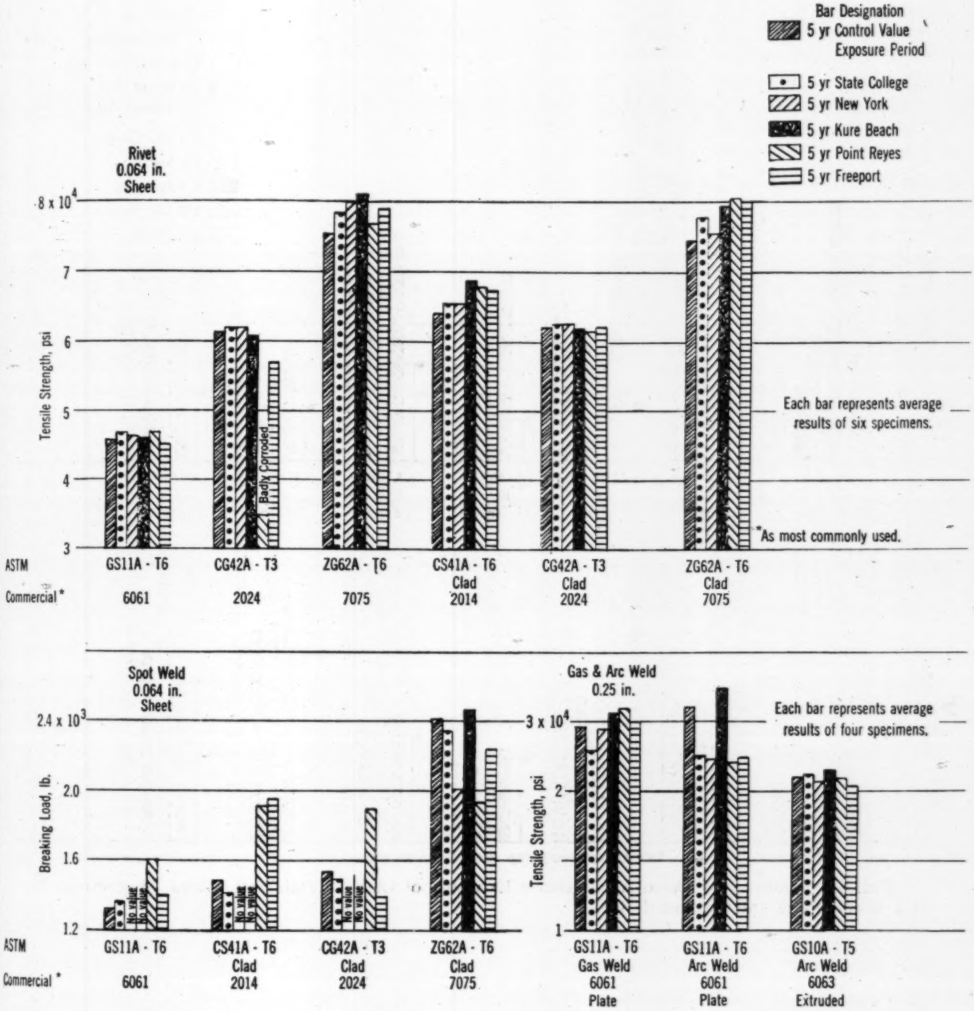
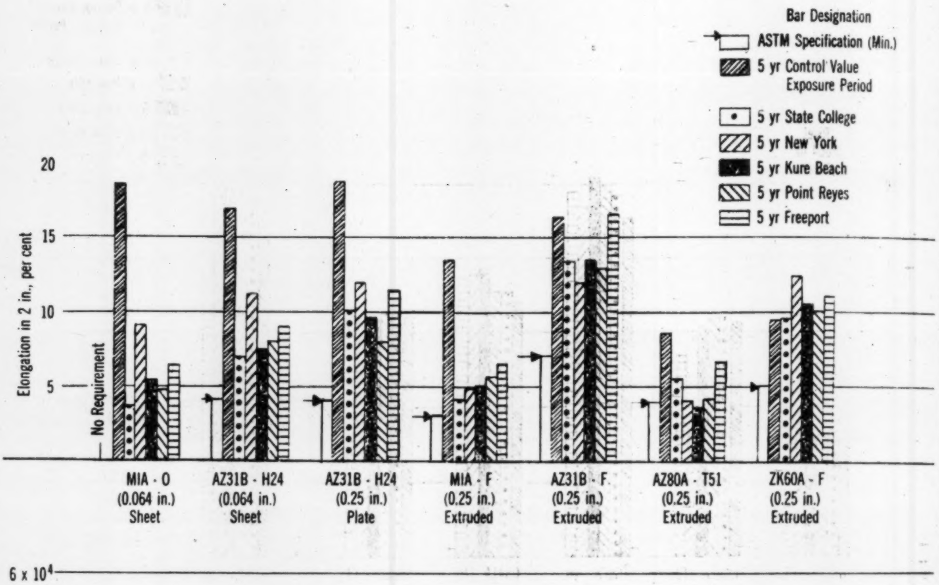


FIG. 3.—Summary of Data, Atmospheric Exposure of Light Metals and Alloys, Aluminum Alloys.



Each bar represents average results of four specimens.

FIG. 4.—Summary of Data, Atmospheric Exposure of Light Metals and Alloys, Magnesium Alloy Sheet, Plate and Extruded Bar.

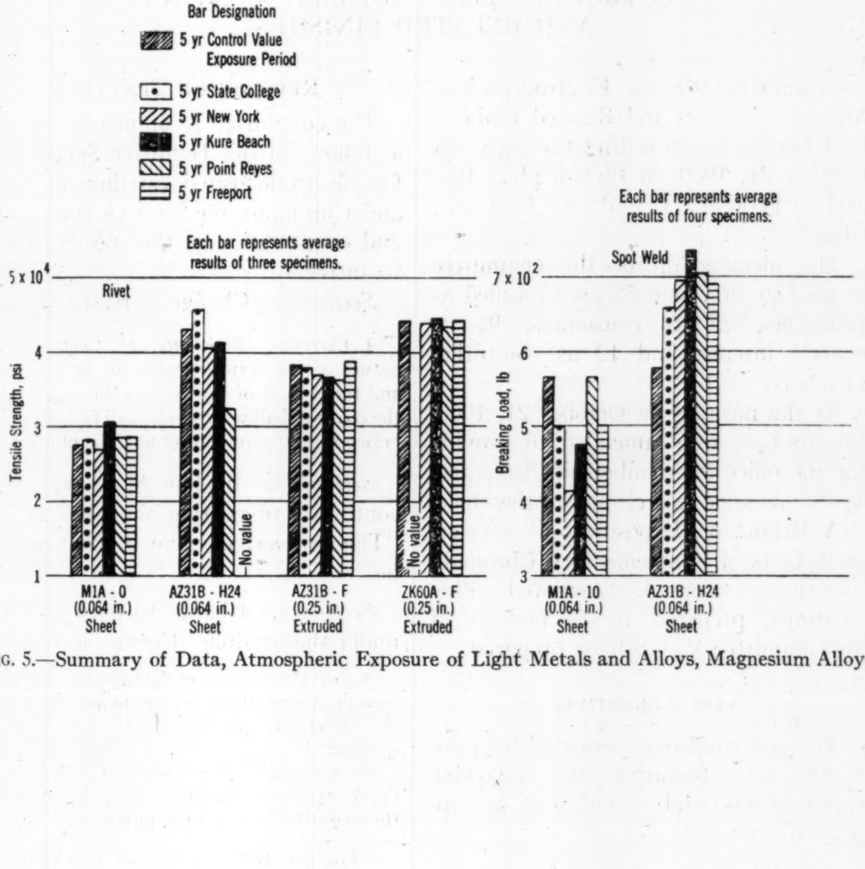


FIG. 5.—**Summary of Data, Atmospheric Exposure of Light Metals and Alloys, Magnesium Alloys.**

REPORT OF COMMITTEE B-8

ON

ELECTRODEPOSITED METALLIC COATINGS AND RELATED FINISHES*

Committee B-8 on Electrodeposited Metallic Coatings and Related Finishes held two meetings during the year: on October 21, 1960, in Philadelphia, Pa., and on February 3, 1961, in Cincinnati, Ohio.

The membership of the committee totals 186, of whom 58 are classified as producers, 25 as consumers, 91 as general interest and 12 as consulting members.

At the meeting on October 21, 1960, the By-laws were amended to provide for the office of Membership Secretary. W. S. Garwood was elected to the office.

A Report on Exposure and Accelerated Tests of Supplementary Chromate Treatments for Electroplated Zinc Coatings, prepared by Section B of Subcommittee V, has been prepared.[1]

NEW TENTATIVES

The committee recommends for publication as tentative the following specifications and definitions as appended hereto.[2]

Tentative Specification for:

Electrodeposited Coatings of Multi-Layer Nickel plus Chromium on Steel.

Tentative Definitions of Terms Relating to:

Electroplating.

REVISION OF TENTATIVE

The committee recommends revisions as follows of the Tentative Specification for Electrodeposited Coatings of Nickel and Chromium on Steel (**A 166 - 58 T**)[3] and continuation of the specification as tentative.

Section 4.—Change to read as follows:

4. *Corrosion Resistance of Coatings.*—The nature of the corrosion tests to be performed, and the periods of exposure to these tests which the coating shall withstand, shall be agreed upon between the manufacturer and the purchaser.

Section 7.—In line 8, change "The continuity of coating shall be" to read "The protective value of coating shall be."

Section 9.—Change to read as follows under the heading "Corrosion Tests:"

9. (*a*) *Preparation of Specimen.*—The specimen shall be prepared for test in accordance with the directions specified in the applicable test method.

(*b*) *Procedure.*—The specimen shall be tested in accordance with methods agreed upon between the manufacturer and the purchaser.[5]

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[5] For suitable methods see the Method of Copper-Accelerated Acetic Acid-Salt Spray (Fog) Testing (Cass Test) (ASTM Designation: B 368) and the Method of Corrosion Testing of Decorative Chromium Plating by the Corrodkote Procedure (ASTM Designation: B 380).

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as follows of the Standard Specifications for Electrodeposited Coatings of Nickel and Chromium on Zinc and Zinc-Base Alloys (**B 142 – 59**)[4] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] See p. 359.

[2] The new tentatives appear in the 1961 Book of ASTM Standards, Part 2.

[3] 1958 Book of ASTM Standards, Part 2.

Section 4.—Revise as recommended above for Section 4 of Specification A 166.

Section 7.—Revise as recommended above for Section 7 of Specification A 166.

Section 9.—Revise as recommended above for Section 9 of Specification A 166.

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Standard Specifications for:

Electrodeposited Coatings of Zinc on Steel (**A 164 – 55**), and
Electrodeposited Coatings of Cadmium on Steel (**A 165 – 55**).

Recommended Practice for Preparation of:

Low-Carbon Steel for Electroplating (**B 183 – 49**),
High-Carbon Steel for Electroplating (**B 242 – 54**),
Zinc-Base Die Castings for Electroplating (**B 252 – 53**),
Electroplating on Aluminum Alloys (**B 253 – 53**), and
Electroplating on Stainless Steel (**B 254 – 53**).

STANDARDS WITH REVISIONS PENDING

The following standards are being continued but without reapproval pending study to resolve several controversial points.

Recommended Practice for:

Chromium Plating on Steel for Engineering Use (**B 177 – 55**).[3] This action is taken to permit balloting in Subcommittee IV on a proposed change in Section 7(b) on Treatments of Chromium Coating as well as a change in a footnote.

Standard Method of Test for:

Dielectric Strength of Anodically Coated Aluminum (**B 110 – 45**)[3], and
Weight of Coating on Anodically Coated Aluminum (**B 137 – 45**).[3]

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Specification for:

Chromate Finishes on Electrodeposited Zinc, Hot-Dipped Galvanized, and Zinc Die-Cast Surfaces (**B 201 – 55 T**).

Tentative Recommended Practice for:

Cleaning Metals Prior to Electroplating (**B 322 – 58 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Papers, Specifications, and Definitions (F. Ogburn, chairman) prepared the Definitions of Terms for Electrodeposited Metallic Coatings and Related Finishes referred to earlier in the report.

Section G (R. B. Saltonstall, chairman) prepared the Specifications for Electrodeposited Coatings of Multi-Layer Nickel Plus Chromium on Steel referred to earlier in the report.

Subcommittee II on Performance Tests (W. H. Safranek, chairman).—The report of Section A on Program No. 4 (Performance of Copper, Nickel, Chromium Coatings) has been prepared.[6]

Section F (Preparation and Corrosion Studies of Plated Coatings on

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[4] 1959 Supplement to Book of ASTM Standards, Part 2.

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[6] See p. 329.

Aluminum) has merged with Section A under the chairmanship of B. B. Knapp.

Subcommittee III on Conformance Tests (A. D. Squitero, chairman).—Section C (M. M. Beckwith, chairman) has completed a report on data obtained on hardness tests.

Subcommittee IV on Electroplating Practice (E. B. Saubestre, chairman).—Section C (B. Cohen, chairman) has completed work on additions to the Recommended Practice for Chromium Plating on Steel for Engineering Use (B 177).

Subcommittee V on Supplementary Treatments for Metallic Coatings (R. E. Harr, chairman).—Section B (A. Mendizza, chairman) has under preparation a Report on Exposure and Accelerated Tests of Supplementary Chromate Treatments for Electroplated Zinc Coatings.[1]

Subcommittee VI on Anodic Oxidation of Aluminum and Magnesium Alloys (R. F. Hafer, chairman) is studying the Standard Method of Test for Dielectric Strength of Anodically Coated Aluminum (B 110 - 45), and Standard Method of Test for Weight of Anodically Coated Aluminum (B 137 - 45) in order to resolve several controversial points.

This report has been submitted to letter ballot of the committee, which consists of 113 voting members; 113 members returned their ballots, of whom 97 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

C. H. SAMPLE,
Chairman.

D. M. BIGGE,
General Secretary.

APPENDIX I

PERFORMANCE OF DECORATIVE NICKEL-CHROMIUM COATINGS ON STEEL

REPORT OF SECTION A, SUBCOMMITTEE II ON PERFORMANCE TESTS

A continuing study of the performance of decorative nickel-chromium coatings on steel has been made under Program No. 4 by Subcommittee II. The principal objectives of this program were to determine: (1) effect of nickel and copper strikes under bright nickel, (2) the effect of type and thickness of chromium, and (3) the effect of buffing bright nickel. Comparison between buffed Watts nickel and bright nickel, and between copper-nickel-chromium and nickel-chromium was also made. Twenty-six lots of panels were plated for this program; exposure was initiated on April 28, 1956, at Kure Beach (800-ft site), N. C., and on May 1, 1956, at Detroit, Mich.

The present report describes preparation of the panels, gives the inspection data up to and including three years exposure and summarizes the results.

Outline of Program:

The combinations and thicknesses of nickel, copper, and chromium plates and other variables included in the program are listed in Table I.

Interest in a nickel strike versus a copper strike under bright nickel motivated the study of their effects on the weathering behavior of the coating. Use of a nickel strike has several advantages. It allows fewer steps in the production cycle and eliminates costly disposal of cyanide wastes. A cursory survey of nickel strikes used in industry indicated that they could be broadly classified into two types: a Watts bath operated at pH 2.0 to 3.5, and a bath at pH 0.5 containing equal amounts of chloride and sulfate.

Within the industry there existed a question as to the optimum thickness of chromium for best performance on bright nickel. Accordingly four thicknesses of chromium from each of two commonly used chromium baths were included in the program. A proprietary dull crack-free chromium deposit over bright nickel and buffed Watts nickel was also tested in two thicknesses.

Buffing of bright nickel is done commercially to some extent to bring up the luster of dull areas. To determine what effect it might have on the performance of the coatings, one lot of panels was bright nickel plated and buffed.

Three thicknesses of bright nickel were tested to serve as a yardstick for determining the extent of the influence of the variables being studied. Two sets of controls were prepared, one at the beginning and one at the end, to check consistency of operation.

Preparation of Test Panels:

The steel for the panels was hot rolled, pickled SAE 950. The surface was flat polished to a roughness of 10 to 12 microinches and then Bonderized. An adhesive-coated paper was used to protect the surface during shipment to the platers. Standard Steel Division of the Rockwell Spring and Axle Co. prepared the steel panels.

The panels were plated by the laboratories of Chrysler Corp., General Motors Corp., The International Nickel Co., Inc., and The United Chromium Division of Metal and Thermit Corp. Two laboratories, hereinafter referred to as plater *X* and plater *Y*, each prepared fifteen panels per lot. A third laboratory designated as plater *Z* prepared fifteen panels of only the lots with buffed Watts nickel. The CR110 and CF500 deposits, the 30- and 60-microinch thick deposits of regular chromium, and the chromium on the panels plated with buffed Watts nickel were plated by United Chromium. The remainder of the chromium plating was done by the plater of the nickel.

TABLE I.—OUTLINE OF PLATING VARIABLES FOR EACH TYPE OF PLATE, THICKNESS IN MILS.

Steel, SAE No. 950

Lot	Strike	Copper	Bright Nickel	Buffed Watts	Regular Cr	CR-110 Cr[a]	CF-500 Cr[a]
No. 1	...	...	1.5	...	0.01	...	...
No. 2	0.05 Cu	...	1.45	...	0.01	...	...
No. 3	0.05 Ni[b]	...	1.45	...	0.01	...	...
No. 4	0.15 Cu	0.65	0.7	...	0.01	...	...
No. 5	0.15 Ni[c]	0.65	0.7	...	0.01	...	...
No. 6	0.15 Ni[c]	...	1.35	...	0.01	...	...
No. 7	0.15 Cu	...	1.35	...	0.01	...	...
No. 8	...	...	1.5	...	0.005	...	...
No. 9	...	...	1.5	...	0.03[a]	...	...
No. 10	...	...	1.5	...	0.06[a]	...	...
No. 11	...	...	1.5	...	...	0.005	...
No. 12	...	...	1.5	...	...	0.01	...
No. 13	...	...	1.5	...	...	0.03	...
No. 14	...	...	1.5	...	...	0.06	...
No. 15	...	...	...	1.5	0.01[a]	...	...
No. 16	...	...	...	1.5	0.03[a]	...	...
No. 17	...	...	...	1.5	0.06[a]	...	...
No. 18	...	...	1.5	...	...	...	0.01
No. 19	...	...	1.5	...	...	...	0.06[d]
No. 20	...	...	1.5[d]	...	0.01	...	...
No. 21	...	...	0.75	...	0.01	...	...
No. 22	...	...	2.5	...	0.01	...	...
No. 23	...	...	1.5	...	0.01	...	...
No. 24	...	...	...	1.5	...	...	0.05[d]
No. 25	...	...	...	1.5	...	...	0.01
No. 26	...	...	...	1.5	0.02[a]	...	...

[a] Chromium was plated by United Chromium. [b] Low pH bath. [c] Watts bath. [d] Buffed.

The sequence of steps in the cleaning and plating cycles for preparing the panels is listed in Table II. The composition and operating conditions for cleaners, pickles, and plating baths are given in Table III.

The bright nickel in lot 20 was buffed by plater *X* using a chromium buffing compound (McAleer D133) on a loose cotton buff (96 × 93 count). A 14-in. wheel at 2300 rpm gave a surface speed of about 8400 ft per min. Plater *Y* used H-VW-M NOCAST lime on a loose leaf cotton buff (64 × 64 count). A 12-in. wheel at 1750 rpm gave a surface speed of about 5500 ft per min. The heavy crack-free chromium plate in lots 19 and 24 was buffed with Matchless Compound No. 525 on a loose sewn cotton buff using a wheel diameter of 12 in. at a speed of 1800 rpm (5600 surface ft per min).

Platers *X* and *Y* processed the panels

TABLE II.—CLEANING AND PLATING CYCLES FOR PREPARATION OF PANELS.

Step	Nickel on Steel	Lot 2	Lots 3 and 6	Lot 4	Lot 5	Lots 15, 16, 17, 20, 24, 25 and 26	Chromium on Nickel, Lots 9–19 and 24–26
No. 1	Anodic clean	Anodic clean	Anodic clean	Anodic clean	Anodic clean	Anodic clean	Cathodic clean
No. 2	Water rinse	Water rinse	Water rinse	Water rinse	Water rinse	Water rinse	Water rinse
No. 3	Acid dip	Acid dip	Acid dip	Acid dip	Acid dip	Acid dip	Cathodic acid clean
No. 4	Water rinse	Water rinse	Water rinse	Water rinse	Water rinse	Water rinse	Water rinse
No. 5	Nickel plate	Cyanide copper	Nickel strike	Cyanide copper	Nickel strike	Nickel plate	Repeat Step 1
No. 6	Water rinse	Water rinse	Nickel plate	Water rinse	Water rinse	Water rinse	Repeat Step 2
No. 7	Chromium plate	Acid dip	Water rinse	Acid dip	Acid copper	Buff nickel	Acid dip
No. 8		Water rinse	Chromium plate	Water rinse	Water rinse	Cathodic clean	Water rinse
No. 9		Nickel plate		Acid copper	Buff copper	Water rinse	Chromium plate
No. 10		Water rinse		Water rinse	Copper cleaner	Acid dip	
No. 11		Chromium plate		Buff copper	Water rinse	Water rinse	
No. 12				Copper cleaner	Acid dip	Chromium plate	
No. 13				Water rinse	Water rinse		
No. 14				Acid dip	Nickel plate		
No. 15				Water rinse	Water rinse		
No. 16				Nickel plate	Chromium plate		
No. 17				Water rinse			
No. 18				Chromium plate			

TABLE III.—COMPOSITION AND OPERATING CONDITIONS FOR CLEANING, PICKLING, AND PLATING BATHS.

A. Anodic Alkaline Cleaner:

Proprietary cleaner—8 oz per gal, 180 F, 60 amp per sq ft, 1 min.

B. Copper Cleaner:

Proprietary cleaner—8 oz per gal, 180 F, 60 amp per sq ft, 1 min.

C. Cathodic Cleaner for Nickel:

Proprietary cleaner—8 oz per gal, 160 to 180 F, 150 to 225 amp per sq ft, 30 sec.

D. Acid Dip:

On buffed nickel and on steel—

Hydrochloric acid (HCl, sp gr 1.16)	50 per cent by vol
Temperature	70 to 80 F
Time	20 sec

After cyanide copper plate—

Sulfuric acid (H_2SO_4, sp gr 1.836)	10 per cent by vol
Temperature	60 to 70 F
Time	5 sec

On nickel in Lots 9 to 19 and 24 to 26—

Sulfuric acid (H_2SO_4, sp gr 1.836)	10 per cent by vol
Temperature	60 to 70 F
Time	5 to 10 sec

E. Cathodic Acid Cleaner:

Sulfuric acid (H_2SO_4, sp gr 1.836)	10 per cent by vol
Temperature	60 to 70 F
Current	200 amp per sq ft
Time	30 sec

F. Cyanide Copper Strike:

Copper (Cu)	4.5 oz per gal
Free cyanide (NaCN)	1.0 oz per gal
Rochelle salts	6.0 oz per gal
Carbonate (Na_2CO_3)	4.6 oz per gal
Temperature	125 to 130 F
Current	25 amp per sq ft

G. Acid Copper (Same as Program No. 3):

Copper sulfate ($CuSO_4 \cdot 5H_2O$)	27 oz per gal
Sulfuric acid (H_2SO_4)	7.8 oz per gal
Addition agents	thiourea type
Temperature	70 to 80 F
Air agitation.	

H. Nickel Strike for Lot 3:

Nickel sulfate ($NiSO_4 \cdot 7H_2O$)	16 oz per gal
Nickel chloride ($NiCl_2 \cdot 6H_2O$)	16 oz per gal
Boric acid (H_3BO_3)	2.5 oz per gal
pH	0.5
Temperature	room
Current	30 amp per sq ft

I. Nickel Strike for Lots 5 and 6:

Nickel sulfate ($NiSO_4 \cdot 7H_2O$)	32 oz per gal
Nickel chloride ($NiCl_2 \cdot 6H_2O$)	8 oz per gal
Boric acid (H_3BO_3)	5 oz per gal
pH	2.5
Temperature	130 F
Current	40 amp per sq ft
Surface tension	40 dynes per cm

J. Watts Nickel:

Nickel sulfate	44 oz per gal
Nickel chloride	6 oz per gal
Boric acid	5 oz per gal
pH	1.8 to 2.0
Surface tension	40 dynes per cm

K. Bright Nickel:

Nickel (Ni)	10.5 oz per gal
Chloride (Cl)	2.6 oz per gal
Boric acid (H_3BO_3)	5 oz per gal
Proprietary brighteners.	
pH	4.0
Temperature	140 F

L. Chromium (Regular):

Chromic acid (CrO_3)	33.3 oz per gal
Sulfate (SO_4)	0.33 oz per gal
Temperature	115 F
Current	1 to 1½ amp per sq in.

M. Chromium (CR 110):

Chromic acid (CrO_3)	30 oz per gal
Sulfate (SO_4)	0.13 oz per gal
Temperature	115 F
Current	¾ to 1 amp per sq in.

N. Chromium (CF-500):

Chromic acid (CrO_3)	36.6 oz per gal
Sulfate (SO_4)	0.13 oz per gal
Temperature	150 F
Current	1½ to 2 amp per sq in.

through the cleaning and nickel plating cycles on racks holding six panels, three abreast and two high. Anodes and racks were arranged to give uniform distribution of deposit over a rack of panels. Plater *Z* plated the panels singly. After chromium plating, the thickness of coating on each panel was measured with a Magne-Gage. These data are filed in the ASTM Headquarters research file RR10:B-8.

Lots 24, 25, and 26 of buffed Watts nickel on a different lot of steel were plated with dull crack-free and regular chromium for purposes of comparison.

Exposure:

One set of five panels from each plater and from each lot was exposed at Kure Beach on April 28, 1956, and at Detroit on May 1, 1956, except that lots 24, 25, and 26 were not exposed at Detroit. The exposures were made on the ASTM-type rack which faces south and holds the panels at an angle of 30 deg from the horizontal at Kure Beach and 45 deg at Detroit. At Kure Beach the exposure was made in the 800-ft lot and at Detroit on the second floor roof of the Chrysler Engineering building. The third set of panels was reserved for accelerated testing.

Inspection:

The panels were inspected individually in the same manner as used in previous programs which consisted in comparing their appearance to rating "standards."[1] Photographic "standards" were employed at the marine exposure, rating charts prepared by Chrysler Corp. at the industrial site. In addition to the appearance rating, a protection rating was also assigned to the panels. In the tabulated data, two rating numbers are given. Both numbers are really based on the appearance of the panels and help to describe more completely the type and extent of deterioration of the coating. The first number, the protective value rating, relates only to the manifestations of basis metal corrosion, that is, readily visible rust and rust stain. The second number, the appearance rating, includes, in addition to the basis metal corrosion and stain, other visible defects in the coating, such as surface pitting, crazing, blisters, etc. It is obvious that the appearance rating cannot be higher than the protection rating; it can, however, be considerably lower. For example, a panel which shows no basis metal corrosion but the surface of which is badly crazed or shows myriads of surface pits may have an appearance rating as low as 1 or 0 even when the protection rating is 10. The condition of the panels is further described by an abbreviated description of the type of defect:

TYPES OF FAILURE

R = Corrosion (rusting) of the basis metal. (Permanent or massive type of basis metal corrosion such as that in pinholes, bare or flaked areas, or in craters of broken blisters.)
R_s = Stain due to basis metal corrosion products, such as rust stain, which can be removed readily with a damp cloth or chamois and mild abrasive revealing a sound bright surface.
S = Stains or spots other than that of *obvious* basis metal corrosion products.
S_p = Surface pits. Corrosion pits probably not extending through to the basis metal—that is, absence of *obvious* basis metal corrosion products bleeding therefrom.
pR = Pin-hole rusting.
F = Flaking or peeling of deposit.
B = Blistering.
C = Cracking.
Z = Crazing.
W = Crow's feet.

DEGREE OF EXTENT OF PINHOLE RUSTING, STAINING, SURFACE PITTING, FLAKING, ETC.

vs = very slight amount.
s = slight amount.
i = intermediate or moderate amount.
x = excessive amount.

DESCRIPTION OF BLISTERS

s = less than about 0.5 mm in diameter.
i = about 0.5 to 2.0 mm in diameter.
x = greater than about 2.0 mm in diameter.
vf = 5 or fewer.
f = 5+ to 25.
i = 10+ to 25.
m = 25+ to 50.
vm = over 50.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 49, p. 226 (1949).

DESCRIPTION OF LOCATION OF DEFECTS

e = edge g = general

EXAMPLES

1. ipR, vscR, sRs = a moderate amount of pin-point-type rusting, a very slight amount of crater-type rusting, and a slight amount of rust stain which can be readily removed.

2. iSp, iS, iiW, seZ = moderate surface pitting, moderate staining (other than rust), an intermediate number of moderate size crow's feet, a small amount of edge crazing.

3. xcR, xRs = excessive crater-type rust spots and an excessive amount of removable rust over a sound, bright surface.

Inspections at Kure Beach were made without cleaning the panels. At Detroit the panels were wet swabbed lightly and blotted dry. Some ratings at Detroit were higher after three years than two years as a result of heavy rain washing away surface stain.

The inspection data to date are summarized in Tables IV and V and the individual panel ratings are given in Tables VI and VII. The average appearance and protection ratings of the panels for 1-, 2-, and 3-yr exposures are shown graphically in Figs. 1 and 2 for Detroit and Kure Beach respectively. Additional inspections after four and seven months were made at Kure Beach because of the rapid deterioration at this exposure. After the 2-yr inspection at Kure Beach, representative panels from each lot and from each plater were removed, along with seven other panels whose performance was erratic. These panels were photographed individually and then checked for thickness of chromium, copper, and nickel. These data are tabulated in Table VIII. The photographs (color transparencies) are filed in the ASTM Headquarters research file RR10:B-8.

The exposure of the panels continues at both sites.

Discussion:

A marked difference in performance of panels plated by platers *X* and *Y* occurred in those lots containing copper in the coating. In addition to possible unknown factors in copper plating being the cause, another explanation relates to the chromium plating. At Kure Beach the panels plated by plater *Y* are consistently better than those plated by plater *X* in lots 1 through 8 and 21 through 23 in which lots each plater chromium plated his own panels. Only in lot 20 are the panels of plater *X* better than plater *Y*. The difference here is small, and the effect of chromium plating may have been outweighed by that of buffing the bright nickel. In lots 9 through 19 there is very little variation between plater *X* and plater *Y* and in these lots all chromium plating was done by the same plater.

Summary of Results:

1. The rate of deterioration of the coatings has been much slower at the Detroit site than at Kure Beach. Therefore the comparisons among lots at Detroit may be less significant.

2. A nickel strike under bright nickel had no effect in the Detroit exposure. A copper strike lowered the rating, its detrimental effect decreasing the rating as much as three rating numbers after three years. A nickel strike was also without effect in the marine atmosphere. A copper strike on panels prepared by plater *X* had no beneficial effect, but on those prepared by plater *Y* the effect was markedly beneficial and the thinner strike was superior.

3. Interposing a layer of acid copper between the strike coatings and the bright nickel had no effect at Detroit. At Kure Beach it had a beneficial effect only with plater *Y*, and here it was more pronounced with the copper than with the nickel strike. Copper in the coating promoted crater-type defects.

4. Comparison of coatings with and without copper under bright nickel indi-

cated that the all-nickel coatings were superior by two to three rating numbers at Detroit after three years. At Kure Beach, the all-nickel coatings had little or no advantage for plater *X*, and for plater *Y* they were markedly inferior to copper-nickel coatings.

5. The thicker chromium coatings of 30 and 60 microinches were more protective on both bright and Watts nickel at Detroit. At Kure Beach the advantage of the thicker chromium on bright nickel was very marked and it was also beneficial on buffed Watts nickel for platers *Y* and *Z*. There was no significant difference between regular and CR-110 chromium, and the buffed crack-free chromium showed no significant benefit. However, all chromium coatings more than 10 microinches in thickness except buffed crack-free chromium exhibited a crazed appearance at both exposure sites.

6. Buffing bright nickel had no significant effect at Detroit, but was very beneficial at Kure Beach. It raised the rating from 0 to 8 for plater *X* and from 0 to 4 for plater *Y*, making it superior to buffed Watts nickel.

7. Watts nickel had a slightly lower appearance rating than bright nickel at Detroit as a result of fine surface pitting. Protection rating was nearly perfect. Watts nickel had a better appearance rating under 10 microinches of chromium at Kure Beach. At greater chromium thicknesses bright nickel was equivalent to buffed Watts nickel.

8. Discrepancies in performance of replicate panels removed after two years at Kure Beach could not be attributed to differences in thickness of copper, nickel, or chromium layers.

9. Good agreement was obtained between platers *X* and *Y* at Detroit on all lots and at Kure Beach on all lots except those containing copper. In this instance the panels plated by plater *Y* were distinctly superior and the cause could not be attributed to thickness. Neither did recognized differences in plating procedure appear causative.

Respectfully submitted,

B. B. KNAPP, *Chairman*,
Section A, Subcommittee II.

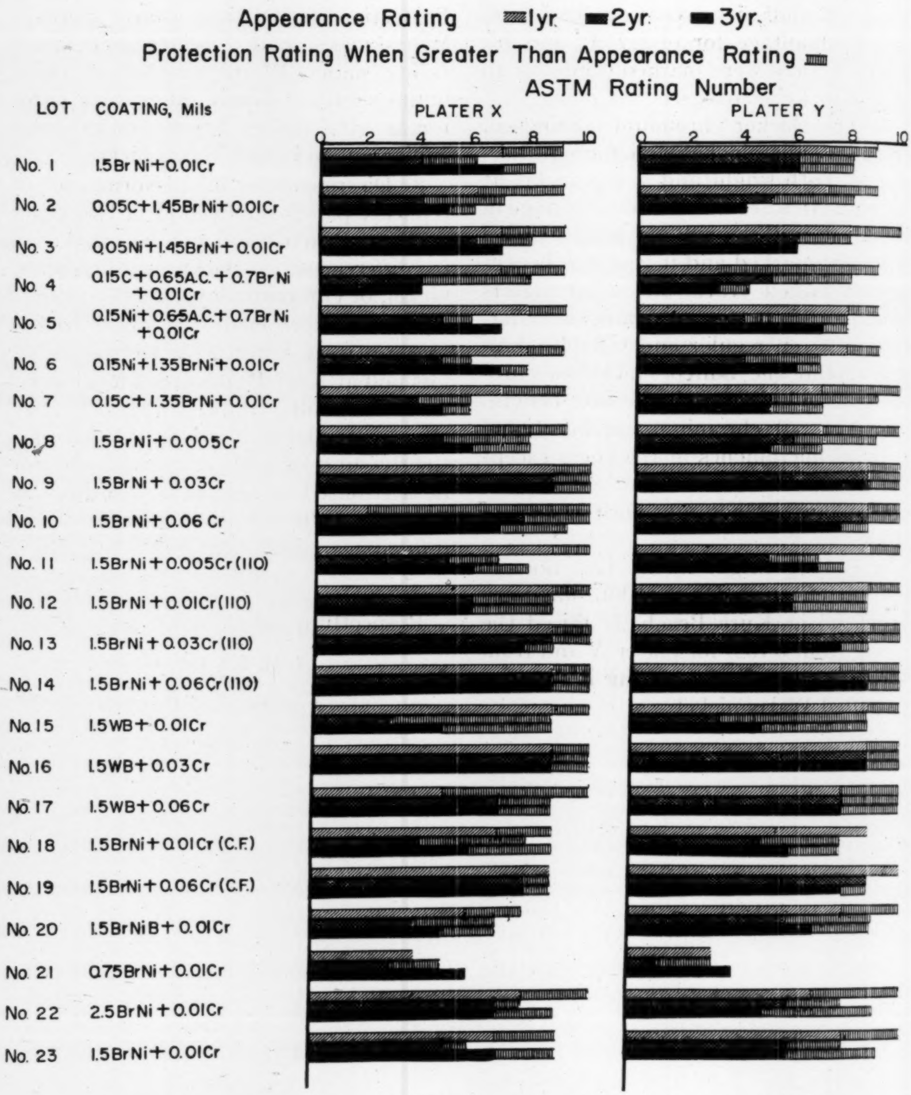

FIG. 1.—Appearance and Protection Ratings of Panels Exposed at Detroit, Mich.

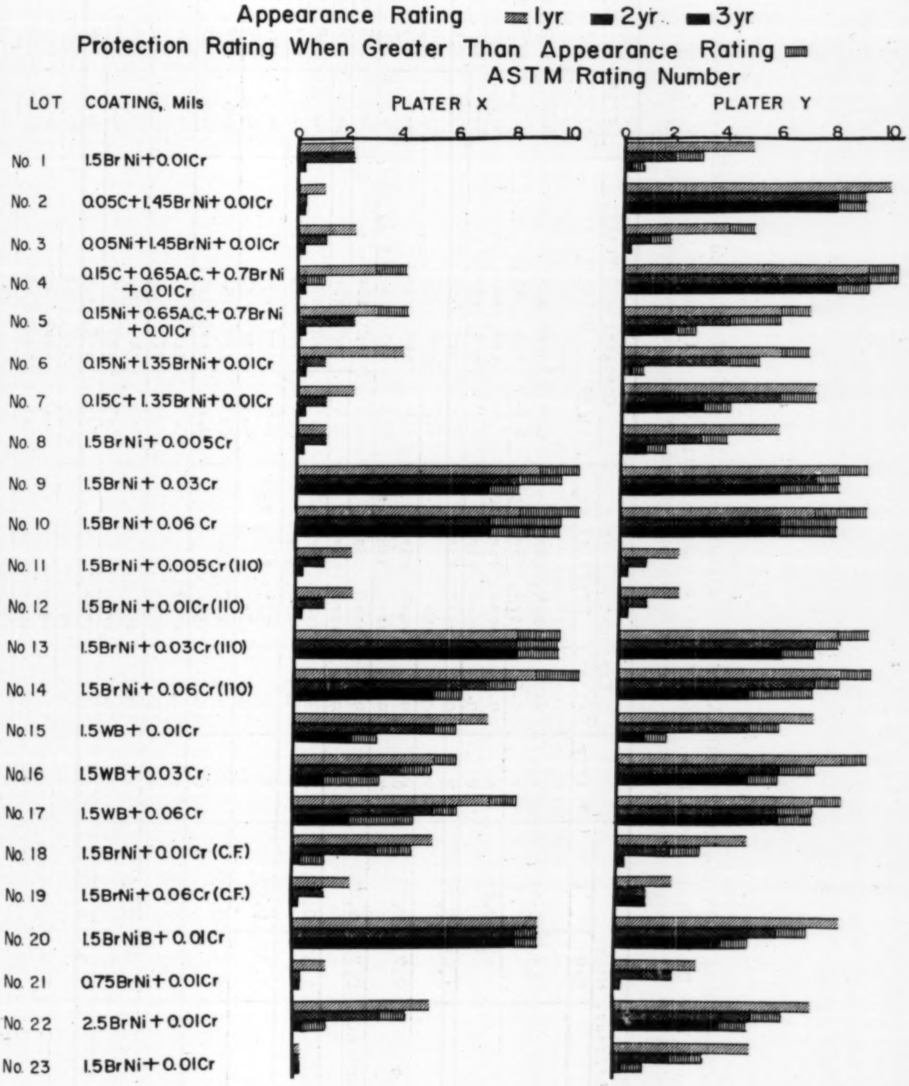

FIG. 2.—Appearance and Protection Ratings of Panels Exposed at Kure Beach N. C.

TABLE IV.—INSPECTION DATA, PROGRAM NO. 4, DETROIT, MICH., EXPOSURE, SUMMARIZING TABLE VI.

ACu = Acid copper, W = Buffed Watts nickel, B = Buffed, CR = Chromium CR 110, CF = Chromium CF 500, CrZ = Chromium crazing, E = Etch. See pages 333 and 334 for explanation of symbols showing descriptions of types of failure, extent, and location of defects.

Lot	Coating, Mils				Plater	Exposure - 1 Year		Exposure - 2 Years		Exposure - 3 Years	
	Strike		BrNi	Cr		Rating	Description	Rating	Description	Rating	Description
1			1.5	0.01	X	9-7	vspR, sSp	6-4	ipR, sSp	8-7	spR, sSp
					Y	9-7	vspR, sSp, ieZ	8-6	vspR, sSp	8-6	spR, iSp
2	0.05 Cu		1.45	0.01	X	9-6	vspR, iSp, iB	7-4	spR, iSp, iB	6-5	ipR, iB, iSp
					Y	9-7	vspR, sSp	8-5	spR, iSp	4-4	xpR, xSp
3	0.05 Ni		1.45	0.01	X	9-7	vspR, sSp	8-6	spR, iSp	7-7	ipR, iRs
					Y	10-8	sSp	8-6	spR, iSp	6-6	ipR, iSp
4	0.15 Cu	0.65 ACu	0.7	0.01	X	9-7	vspR, sSp	8-4	spR, xSp	4-4	iB, icR
					Y	9-3	vspR, vxSp, seZ	8-5	spR, xSp, seZ, vsW	4-3	xpR, iSp, xB
5	0.15 Ni	0.65 ACu	0.7	0.01	X	9-8	vspR, sSp	6-5	ipR, sSp	7-7	ipR, vsSp
					Y	9-8	vspR, sSp	8-4	spR, xSp	8-7	spR, iSp
6	0.15 Ni		1.35	0.01	X	9-8	vspR, sSp, sRs	6-5	ipR, sSp	8-7	spR, iSp
					Y	9-8	vspR, sSp	7-4	ipR, xSp	7-6	spR, sSp
7	0.15 Cu		1.35	0.01	X	9-7	vspR, sSp, vsB	6-4	ipR, sSp, iB	6-5	iB, iSp
					Y	10-7	sSp	9-5	vspR, xSp	7-5	ipR, iSp
8			1.5	0.005	X	9-6	vspR, iSp	8-5	spR, xSp	8-6	spR, iSp
					Y	10-7	iSp	9-6	vspR, xSp	7-5	ipR, iSp
9			1.5	0.03	X	10-9	sCrZ	10-9	sZ	10-9	sZ
					Y	10-9	sCrZ	10-9	sZ	10-9	sZ
10			1.5	0.06	X	10-2	xE	10-8	iE	9-7	vspR, iE
					Y	10-9	vsE	10-9	vsE	9-8	vspR, sE
11			1.5	0.005 CR	X	10-9	vsSp	7-5	ipR, iSp	8-6	spR, iSp
					Y	10-9	vsSp	7-5	ipR, iSp	8-7	spR, sSp
12			1.5	0.01 CR	X	10-9	vsSp	9-6	vspR, xSp	9-6	vspR, iSp
					Y	10-9	vsSp	9-6	vspR, xSp	9-6	vspR, iSp

13			1.5	0.03 CR	X	10-9	vsSp, vsCrZ	10-9	iZ	9-8	vspR, sZ
					Y	10-9	vspR, vsCrZ	10-9	iZ	9-8	vspR, sZ
14			1.5	0.06 CR	X	10-9	vsE	10-9	vsE	10-9	sZ
					Y	10-8	vsE	10-9	sE	10-9	sZ
15		1.5 W		0.01	X	10-9	vsSp	9-3	vspR, xSp	9-5	vspR, xSp, vsZ
					Y	10-9	vsSp	9-3	vspR, xSp	9-5	vspR, xSp, vsZ
					Z	10-9	vsSp	6-3	ipR, xSp	9-5	vspR, xSp, vsZ
16		1.5 W		0.03	X	10-9	vsCrZ	10-9	vsZ	10-9	vsSp, vsZ
					Y	10-9	vsCrZ, seZ	10-9	vsZ	10-9	vsSp, vsZ
					Z	10-9	vsCrZ, seZ	10-9	vsZ	9-7	vsSp, vspR, iZ
17		1.5 W		0.06	X	10-5	xE, xsS	9-7	vspR, sE	9-7	vspR, sSp, iZ
					Y	10-8	sE	10-8	sE	10-8	sSp, iZ
					Z	10-5	xE	10-8	sE	9-7	vspR, sSp, iZ
18			1.5	0.01 CF	X	9-7	vspR, ssS	8-4	spR, xSp	9-6	vspR, xSp
					Y	9-9	vspR, seR	8-5	spR, xSp	8-6	spR, iSp
19			1.5	0.06 CFb	X	9-9	vspR	9-8	vspR, vsSp	9-8	vspR, sSp
					Y	10-10		9-9	vspR	9-8	vspR, sSp
20			1.5b	0.01	X	8-6	vspR, ssS	7-4	ipR, xSp	7-5	ipR, iSp
					Y	10-8	vsSp	9-6	vspR, xSp	9-7	vspR, iSp
21			0.75	0.01	X	4-4	xpR, xRs	5-3	xpR, xRs, seZ	6-6	ipR, seZ
					Y	3-3	xpR, xRs	3-1	xpR, xRs, seZ	4-4	xpR, seZ
22			2.5	0.01	X	10-8	sSp	8-7	spR, sSp	9-7	vspR, iSp, ieZ
					Y	10-7	sSp	8-6	spR, iSp	9-5	vspR, xSp
23			1.5	0.01	X	9-7	vspR, sSp	6-5	ipR, sSp	9-7	vspR, iSp
					Y	10-8	sSp, ieZ, seR	8-6	spR, iSp	9-6	vspR, xSp

TABLE V.—INSPECTION DATA, PROGRAM NO. 4, KURE BEACH (800-FT SITE), N. C., EXPOSURE, SUMMARIZING TABLE VII.

ACu = Acid copper, W = Buffed Watts nickel, B = Buffed, CR = Chromium CR 110, CF = Chromium CF 500, CrZ = Chromium crazing, E = Etch. See pages 333 and 334 for explanation of symbols showing descriptions of types of failure, extent, and location of defects.

	Coating, Mils					Exposure - 4 Months		Exposure - 7 Months	
Lot	Strike		BrNi	Cr	Plater	Rating	Description	Rating	Description
1			1.5	0.01	X	6-6	iPR	3-3	iPR, icR, iRs
					Y	8-8	sPR	6-6	iPR, icR, iRs
2	0.05 Cu		1.45	0.01	X	3-3	iPR	2-2	xcR, xRs, xPR
					Y	9-9	vsPR	10-9	vsPR
3	0.05 Ni		1.45	0.01	X	9-9	sPR	3-3	iPR, icR, iRs
					Y	9-9	sPR	7-6	iPR, scR, sRs, xvsSP
4	0.15 Cu	0.65 ACu	0.7	0.01	X	7-7	iPR	6-5	iPR, scR, iRs, xvsSP
					Y	10-9	iSP	10-9	xvsSP
5	0.15 Ni	0.65 ACu	0.7	0.01	X	5-5	xPR	3-2	xPR, iRs, xvsSP
					Y	9-9	vsPR	7-7	iPR, iRs, xvsSP
6	0.15 Ni		1.35	0.01	X	6-6	iPR	6-5	iPR, iRs, xvsSP
					Y	7-7	sPR	7-6	iPR, iRs, xvsSP
7	0.15 Cu		1.35	0.01	X	6-6	iPR	4-4	xcR, xRs, iPR
					Y	9-8	sPR, iSP	8-8	iPR
8			1.5	0.005	X	4-4	xPR	2-2	xPR, xRs
					Y	7-7	sPR	7-7	sPR, sRs
9			1.5	0.03	X	9-9	vsPR, xZ	10-9	vsZ
					Y	8-8	sPR, xZ	9-8	vsZ, vsPR
10			1.5	0.06	X	10-10	xZ	10-5	vsZ, xE
					Y	9-9	sPR, xZ	9-6	vsPR, sZ
11			1.5	0.005 CR	X	8-8	sPR	5-5	iPR, iRs
					Y	6-6	iPR, sSP	4-3	iPR, iRs, xvsSP
12			1.5	0.01 CR	X	6-4	iPR, iSP	3-3	iPR, ivsSP, iRs
					Y	6-4	iPR, iSP	4-3	xPR, ivsSP, xRs
13			1.5	0.03 CR	X	9-9	vsPR, xZ	9-8	vsPR, iZ, sE
					Y	9-9	vsPR, xZ	9-7	vsPR, iZ
14			1.5	0.06 CR	X	10-10	xZ	10-9	sZ
					Y	8-8	sPR, xZ	9-8	vsPR, sZ
15		1.5 W		0.01	X	9-9	vsPR	9-9	vsPR
					Y	9-9	vsPR	9-9	vsPR
					Z	9-9	vsPR	9-9	vsPR
16		1.5 W		0.03	X	9-9	vsPR, xZ	9-8	vsPR, sZ
					Y	9-9	vsPR, xZ	9-8	vsPR, sZ
					Z	9-9	vsPR, xZ	9-8	vsPR, sZ
17		1.5 W		0.06	X	9-9	vsPR, xZ	9-6	vsPR, sSP, sE
					Y	9-9	vsPR, xZ	9-8	vsPR, vsSP, sZ
					Z	9-9	vsPR, xZ	9-7	vsPR, sZ, sE
18			1.5	0.01 CF	X	9-9	vsPR	9-9	vsPR
					Y	7-7	sPR	7-7	sPR, iRs
19			1.5	0.06 CFB	X	2-2	xPR, iRs	2-2	xPR, xRs
					Y	5-5	xPR	2-2	xPR, xRs
20			1.5 B	0.01	X	9-9	vsPR	9-9	vsPR
					Y	9-9	vsPR	8-8	vsPR
21			0.75	0.01	X	1-1	xPR, xRs	2-2	xPR, icR, xRs
					Y	1-1	xPR, iRs	4-4	iPR
22			2.5	0.01	X	9-9	vsPR, iSP	9-8	sPR, sSP
					Y	9-9	vsPR, iSP	9-9	vsPR
23			1.5	0.01	X	4-4	xPR	2-1	xPR, xRs, xSP
					Y	9-9	vsPR	8-8	sPR
24		1.5 W		0.05 CFB	Z	6-6	iPR	4-4	xscR, iRs
25		1.5 W		0.01 CF	Z	9-9	vsPR	10-10	
26		1.5 W		0.02	Z	10-10		9-9	vsPR

TABLE V.—*Continued.*

EXPOSURE - 1 YEAR		EXPOSURE - 2 YEARS		EXPOSURE - 3 YEARS	
RATING	DESCRIPTION	RATING	DESCRIPTION	RATING	DESCRIPTION
2-2	xcR, xpR, xRs	2-2	xpR, xRs	0-0	xpR, xRs, icR
5-5	iRs, ipR, iEZ	3-2	xpR, xRs, iSp	1-0	xpR, xRs
1-1	xcR, xpR, xRs	0-0	xcR, xB	0-0	-
10-10		9-8	vspR	9-8	vspR, sSp
2-2	xpR, xRs	1-1	xpR, xRs	0-0	vxpR, xRs
5-4	ipR, iRs, isB, imB	2-1	xpR, xRs, iSp	0-0	vxB, vxpR, xRs
4-3	xcR, iRs, xsP	1-0	vxcR, xRs	0-0	vxcR
10-9	sSp	10-9	sSp	9-8	spR, sSp
4-3	scR, xpR, xRs, iSp	2-2	xpR, icR, xRs	0-0	xcR, xpR, xRs
7-6	spR, vscR, sSp	6-4	xcR, ipR, iRs, iSp	3-2	ipR, sSp, iRs, icR
4-4	ipR, iRs	1-1	xpR, xRs, sSp, scR	0-0	vxpR, xRs
7-6	iSP, spR, scR	5-4	ipR, sRs, sSp	1-0	xpR, xRs
2-2	xcR, iRs	1-1	vxcR, xB, xRs	0-0	vxB, vxcR
7-7	spR,	7-6	ipR, iRs, iSp	4-3	spR, iSp, iRs, scR
1-1	xpR, xRs	1-1	vxpR, xRs	0-0	xpR, xRs
6-6	spR, sRs, iSp	4-3	ipR, sSp	2-1	xpR, xRs
10-9	CrZ	9-8	vspR, iZ	8-7	spR, sE
9-8	vspR, cRz	8-7	spR, iZ	8-6	spR, sE
10-8	sE	9-7	spR, xE	9-7	spR, iE
9-7	vspR, sE	8-6	spR, xE	8-6	spR, iE
2-2	xpR, xRs	1-1	vxpR, xRs,sSp	0-0	vxpR, xRs
2-2	xpR, xRs	1-1	vxpR, xRs, sSp	0-0	vxpR, xRs
2-2	xpR, xRs	1-1	vxpR, xRs, sSp	0-0	vxpR, xRs
2-2	xpR, xRs	1-1	vxpR, xRs, sSp	0-0	vxpR, xRs
9-8	vspR, sE	9-8	spR, sZ	9-8	spR, sE
9-8	vspR, sE	8-7	spR, sZ	7-6	ipR, sE
10-9	vspR, sE	8-7	spR, sZ	6-5	ipR, iE
9-8	vspR, sE	8-7	spR, sZ	7-5	ipR, iE
7-7	ipR	6-5	ipR, iRs, sSp	3-2	xpR, sRs, sSp
7-7	spR	6-5	ipR, iRs, sSp	2-1	xpR, sRs, sSp
8-8	spR	6-5	ipR, iRs, sSp	3-2	xpR, xRs
6-5	ipR, sE	5-4	xpR, sZ	3-1	xpR, xRs, sE
9-8	vspR, sE	7-6	spR, sZ	6-5	ipR, sE
9-8	vspR, sE	9-8	spR, sZ	9-8	vspR, sE
8-7	vspR, sE	6-5	ipR, sZ	4-2	ipR, iRs, sE
8-7	vspR, sE	7-6	spR, sZ	7-6	ipR, iRs, sE
10-9	vspR, sE	8-6	spR, sZ	8-6	ipR, iRs, sE
5-5	ipR	4-3	spR, sSp	1-0	xpR, xRs
5-5	ipR	3-2	xpR, xRs, sSp	0-0	vxpR, vxRs
2-2	xpR	1-1	xpR, icR	0-0	vxpR, xRs
2-2	xpR	1-1	xpR, icR	1-1	vxpR, xRs
9-9	vspR	9-8	spR, sSp	9-8	vspR, vsSp
8-8	spR	7-6	ipR, sSp	5-4	ipR, iRs, iSp
1-1	xpR	0-0	vxpR, xcR	0-0	vxcR, xRs
3-3	ipR	2-2	xpR, scR, sSp	0-0	vxpR, xRs
5-5	ipR	4-3	xpR, sSp	1-0	vxpR, xRs
7-7	spR	6-5	ipR, sSp	5-4	ipR, iRs, sSp
0-0	xpR	0-0	vxpR	0-0	xcR, xpR, xRs
5-5	ipR	3-2	xpR, sSp	1-0	xpR, xRs
2-2	xpR	1-1	vxcR, xB, xRs	0-0	vxcR, xB, xRs
9-9	vspR	9-9	spR	7-5	spR, sSp
9-9	vspR	8-7	spR, sSp	7-6	ipR, sRs, sSp

TABLE VI.—INSPECTION DATA, PROGRAM NO. 4, DETROIT, MICH., EXPOSURE.

ACu = Acid copper, W = Buffed Watts nickel, B = Buffed, CR = Chromium CR 110, CF = Chromium CF 500, CrZ = Chromium crazing, E = Etch. See pages 333 and 334 for explanation of symbols showing descriptions of types of failure, extent, and location of defects.

Lot	Coating, Mils				Plater	Exposure - 1 Year		Exposure - 2 Years		Exposure - 3 Years	
	Strike		BrNi	Cr		Rating	Description	Rating	Description	Rating	Description
1			1.5	0.01	X	9-7	vspR, sSp	6-4	lpR, sSp	8-7	spR, sSp
						9-7	vspR, sSp	7-5	lpR, sSp	8-7	spR, sSp
						9-7	vspR, sSp	6-4	lpR, sSp	8-7	spR, sSp
						9-7	vspR, sSp	6-4	lpR, sSp	8-7	spR, sSp
						9-7	vspR, sSp	6-4	lpR, sSp	8-7	spR, sSp
					Y	9-7	vspR, sSp, lEZ	9-7	vspR, sSp	8-6	spR, lSp
						9-7	vspR, sSp, lEZ	8-6	vspR, sSp	8-6	spR, lSp
						9-7	vspR, sSp, lEZ	9-7	vspR, sSp	8-6	spR, lSp
						9-7	vspR, sSp, lEZ	9-7	vspR, sSp	8-6	spR, lSp
						9-7	vspR, sSp, lEZ	7-5	vspR, sSp	8-6	spR, lSp
2	0.05 Cu		1.45	0.01	X	9-6	vspR, lSp, lB	8-4	spR, lSp, lB	6-5	lpR, lB, lSp
						9-5	vspR, lSp, xB	7-4	spR, lSp, lB	6-5	lpR, lB, lSp
						9-6	vspR, lSp, lB	8-5	spR, lSp, lB	7-6	lpR, lB, lSp
						9-6	vspR, lSp, lB	7-4	spR, lSp, lB	6-5	lpR, lB, lSp
						9-6	vspR, lSp, lB	7-4	spR, lSp, xB	6-5	lpR, lB, lSp
					Y	9-7	vspR, sSp	7-5	spR, lSp	4-4	xpR, xSp
						9-7	vspR, sSp	8-4	spR, lSp, sB	5-4	lpR, xSp, sB
						9-7	vspR, sSp	7-5	spR, lSp,	4-4	xpR, xSp, sB
						9-7	vspR, sSp	8-6	spR, lSp	4-4	xpR, xSp
						9-7	vspR, sSp	9-7	spR, lSp	4-4	xpR, xSp
3	0.05 Ni		1.45	0.01	X	9-7	vspR, sSp	8-6	spR, lSp	7-7	lpR, lRs
						9-7	vspR, sSp	8-6	spR, lSp	7-7	lpR, lRs
						9-7	vspR, sSp	8-6	spR, lSp	7-7	lpR, lRs
						9-7	vspR, sSp	8-6	spR, lSp	7-7	lpR, lRs
						9-7	vspR, sSp	8-6	spR, lSp	7-7	lpR, lRs
					Y	10-8	sSp	8-6	spR, lSp	7-6	lpR, lSp
						10-8	sSp	7-5	spR, lSp	4-4	xpR, lSp
						10-8	sSp	8-6	spR, lSp	7-6	lpR, lSp
						10-8	sSp	8-6	spR, lSp	7-6	lpR, lSp
						10-8	sSp	8-6	spR, lSp	7-6	lpR, lSp

4	0.15 Cu	0.65 ACu	0.7	0.01	X	9-7	vsPR, sSp	8-4	sPR, xSp	4-4	IB, IcR
						9-7	vsPR, sSp	8-4	sPR, xSp	4-4	IB, IcR
						9-7	vsPR, sSp	8-4	sPR, xSp	4-4	IB, IcR
						9-7	vsPR, sSp	8-4	sPR, xSp	4-4	IB, IcR
						9-7	vsPR, sSp	8-4	sPR, xSp	4-4	IB, IcR
					Y	9-3	vsPR, vxSp, sEZ	9-5	sPR, xSp, sEZ, vsW	4-3	xPR, ISp, xB
						9-3	vsPR, vxSp, sEZ	9-5	sPR, xSp, sEZ, vsW	4-3	xPR, ISp, xB
						9-2	vsPR, vxSp, sEZ	8-5	sPR, xSp, sEZ, vsW	4-3	xPR, ISp, xB
						9-3	vsPR, vxSp, sEZ	7-4	sPR, xSp, sEZ, vsW	4-3	xPR, ISp
						9-4	vsPR, xSp, sEZ	8-5	sPR, xSp, sEZ, vsW	5-4	IPR, ISp
5	0.15 Ni	0.65 ACu	0.7	0.01	X	9-8	vsPR, sSp	5-4	IPR, sSp	7-7	IPR, vsSp
						9-8	vsPR, sSp	6-5	IPR, sSp	7-7	IPR, vsSp
						9-8	vsPR, sSp	6-5	IPR, sSp	7-7	IPR, vsSp
						9-8	vsPR, sSp	6-5	IPR, sSp	7-7	IPR, vsSp
						9-8	vsPR, sSp	6-5	IPR, sSp	7-7	IPR, vsSp
					Y	9-8	vsPR, sSp	8-4	sPR, xSp	8-7	sPR, ISp
						9-8	vsPR, sSp	9-4	sPR, xSp	8-7	sPR, ISp
						9-8	vsPR, sSp	9-4	sPR, xSp	8-7	sPR, ISp
						9-8	vsPR, sSp	8-4	sPR, xSp	8-7	sPR, ISp
						9-8	vsPR, sSp	9-4	sPR, xSp	8-7	sPR, ISp
6	0.15 Ni		1.35	0.01	X	9-8	vsPR, sRs, sSp	6-5	IPR, sSp	8-7	sPR, ISp
						9-8	vsPR, sRs, sSp	6-5	IPR, sSp	8-7	sPR, ISp
						9-8	vsPR, sRs, sSp	6-5	IPR, sSp	8-7	sPR, ISp
						9-8	vsPR, sRs, sSp	6-5	IPR, sSp	8-7	sPR, ISp
						9-8	vsPR, sRs, sSp	6-5	IPR, sSp	8-7	sPR, ISp
					Y	9-8	vsPR, sSp	7-4	IPR, xSp	8-6	sPR, sSp
						9-8	vsPR, sSp	7-4	IPR, xSp	5-4	IPR, IRs, sSp
						9-8	vsPR, sSp	7-4	IPR, xSp	8-6	sPR, sSp
						9-8	vsPR, sSp	7-4	IPR, xSp	8-6	sPR, sSp
						9-8	vsPR, sSp	7-4	IPR, xSp	8-6	sPR, sSp

TABLE VI.—*Continued.*

Lot	Coating, Mils				Plater	Exposure - 1 Year		Exposure - 2 Years		Exposure 3 - Years	
	Strike		BrNi	Cr		Rating	Description	Rating	Description	Rating	Description
7	0.15 Cu		1.35	0.01	X	9-7	vsPR, sSp	6-4	lPR, sSp, lB	6-5	lB, lSp
						9-7	vsPR, sSp, vsB	6-4	lPR, sSp, lB	6-5	lB, lSp
						9-6	vsPR, sSp, lB	6-4	lPR, sSp, lB	5-4	lB, lSp
						9-7	vsPR, sSp, sB	6-4	lPR, sSp, lB	6-5	lB, lSp
						9-7	vsPR, sSp	6-4	lPR, sSp, lB	6-5	lB, lSp
					Y	10-7	sSp	9-5	vsPR, xSp	7-5	lPR, lSp
						10-7	sSp	8-4	vsPR, xSp, sB	7-5	lPR, sB, lSp
						10-7	sSp	9-5	vsPR, xSp	8-5	sPR, lSp
						9-7	vsPR, sSp	8-4	vsPR, xSp	6-4	lPR, lRs, lSp
						10-7	sSp	9-5	vsPR, xSp	8-5	sPR, lSp
8			1.5	0.005	X	9-6	vsPR, lSp	8-5	sPR, xSp	8-6	sPR, lSp
						9-6	vsPR, lSp	7-4	sPR, xSp	8-6	sPR, lSp
						9-6	vsPR, lSp	8-5	sPR, xSp	8-6	sPR, lSp
						9-6	vsPR, lSp	8-5	sPR, xSp	8-6	sPR, lSp
						9-6	vsPR, lSp	8-5	sPR, xSp	8-6	sPR, lSp
					Y	10-7	lSp	9-6	vsPR, xSp	7-5	lPR, lSp
						10-7	lSp	9-6	vsPR, xSp	7-5	lPR, lSp
						10-7	lSp	9-6	vsPR, xSp	7-5	lPR, lSp
						10-7	lSp	9-6	vsPR, xSp	7-5	lPR, lSp
						10-7	lSp	9-6	vsPR, xSp	7-5	lPR, lSp
			1.5	0.03	X	10-9	sCrZ	10-9	sZ	10-9	sZ
						10-9	sCrZ	10-9	sZ	10-9	sZ
						10-9	sCrZ	10-9	sZ	10-9	sZ
						10-9	sCrZ	10-9	sZ	10-9	sZ
						10-9	sCrZ, seS	10-9	sZ	10-9	sZ
					Y	10-9	sCrZ	10-9	sZ	10-9	sZ
						10-9	sCrZ	10-9	sZ	10-9	sZ
						10-9	sCrZ, seZ	10-9	sZ	10-9	sZ
						10-9	sCrZ	10-9	sZ	10-9	sZ
						10-9	sCrZ	10-9	sZ	10-9	sZ

10			1.5	0.06	X	10-2	xCrE	10-8	lCrE	9-7	vspR, lE
						10-2	xCrE	10-8	lCrE	9-7	vspR, lE
						10-2	xCrE	10-8	lCrE	9-7	vspR, lE
						10-2	xCrE	9-7	lCrE	9-7	vspR, lE
						10-2	xCrE	10-8	lCrE	9-7	vspR, lE
					Y	10-9	vsCrE	10-9	vsCrE	9-8	vspR, sE
						10-9	vsCrE	10-9	vsCrE	9-8	vspR, sE
						10-9	vsCrE	10-9	vsCrE	9-8	vspR, sE
						10-9	vsCrE	10-9	vsCrE	9-8	vspR, sE
						10-9	vsCrE	10-9	vsCrE	9-8	vspR, sE
11			1.5	0.005 CR	X	10-9	vsSp	7-5	lpR, lSp	8-6	spR, lSp
						10-9	vsSp	7-5	lpR, lSp	8-6	spR, lSp
						10-9	vsSp	7-5	lpR, lSp	8-6	spR, lSp
						10-7	vsSp, lsS	6-4	lpR, lSp	8-6	spR, lSp
						10-9	vsSp	7-5	lpR, lSp	8-6	spR, lSp
					Y	10-9	vsSp	8-6	lpR, lSp	8-7	spR, sSp
						10-9	vsSp	7-5	lpR, lSp	8-7	spR, sSp
						10-9	vsSp	7-5	lpR, lSp	8-7	spR, sSp
						10-9	vsSp	7-5	lpR, lSp	8-7	spR, sSp
						10-9	vsSp	8-6	lpR, lSp	8-7	spR, sSp
12			1.5	0.01 CR	X	10-9	vsSp	9-6	vspR, xSp	9-6	vspR, lSp
						10-9	vsSp	9-6	vspR, xSp	9-6	vspR, lSp
						10-9	vsSp	9-6	vspR, xSp	9-6	vspR, lSp
						10-9	vsSp	9-6	vspR, xSp	9-6	vspR, lSp
						10-9	vsSp	9-6	vspR, xSp	9-6	vspR, lSp
					Y	10-9	vsSp	9-6	vspR, xSp	9-6	vspR, lSp
						10-9	vsSp	9-6	vspR, xSp	9-6	vspR, lSp
						10-9	vsSp	9-6	vspR, xSp	9-6	vspR, lSp
						10-9	vsSp	9-6	vspR, xSp	9-6	vspR, lSp
						10-9	vsSp	9-6	vspR, xSp	9-6	vspR, lSp

TABLE VI.—*Continued.*

Lot	Coating, Mils				Plater	Exposure - 1 Year		Exposure - 2 Years		Exposure - 3 Years	
	Strike		BrNi	Cr		Rating	Description	Rating	Description	Rating	Description
13			1.5	0.03 Cr	X	10-9	vsSp, vsCrZ	10-9	iZ	9-8	vsPR, sZ
						10-9	vsSp, vsCrZ	10-9	iZ	9-8	vsPR, sZ
						10-9	vsSp, vsCrZ	10-9	iZ	9-8	vsPR, sZ
						10-9	vsSp, vsCrZ	10-9	iZ	9-8	vsPR, sZ
						10-9	vsSp, vsCrZ	10-9	iZ	9-8	vsPR, sZ
					Y	10-8	vsPR, vsCrZ	10-9	iZ	9-8	vsPR, sZ
						10-8	vsPR, vsCrZ	10-9	iZ	9-8	vsPR, sZ
						10-9	vsPR, vsCrZ	10-9	iZ	9-8	vsPR, sZ
						10-9	vsPR, vsCrZ, seZ	10-9	iZ	9-8	vsPR, sZ
						10-9	vsPR, vsCrZ	10-9	iZ	9-8	vsPR, sZ
14			1.5	0.06 Cr	X	10-8	sCrE, ssS	10-9	vsCrE	10-9	sZ
						10-8	sCrE, ssS	10-9	vsCrE	10-9	sZ
						10-9	vsE	10-9	vsCrE	10-9	sZ
						10-9	vsE	10-9	vsCrE	10-9	sZ
						10-9	vsE	10-9	vsCrE	10-9	sZ
					Y	10-7	sCrE	10-8	sCrE	10-9	sZ
						10-9	vsE	10-8	sCrE	10-9	sZ
						10-8	sE	10-8	sCrE	10-9	sZ
						10-9	vsE	10-8	sCrE	10-9	sZ
						10-9	vsE	10-8	sCrE	10-9	sZ
15		1.5 W		0.01	X	10-9	vsSp	9-3	vsPR, xSp	9-5	vsPR, xSp, vsZ
						10-9	vsSp	9-3	vsPR, xSp	9-5	vsPR, xSp, vsZ
						10-9	vsSp	8-3	vsPR, xSp	9-5	vsPR, xSp, vsZ
						10-9	vsSp	9-3	vsPR, xSp	9-5	vsPR, xSp, vsZ
						10-9	vsSp	8-3	vsPR, xSp	9-5	vsPR, xSp, vsZ
					Y	10-9	vsSp	9-3	vsPR, xSp	9-5	vsPR, xSp, vsZ
						10-9	vsSp	9-3	vsPR, xSp	9-5	vsPR, xSp, vsZ
						10-9	vsSp	9-3	vsPR, xSp	9-5	vsPR, xSp, vsZ
						10-9	vsSp	9-3	vsPR, xSp	9-5	vsPR, xSp, vsZ
						10-9	vsSp	9-3	vsPR, xSp	9-5	vsPR, xSp, vsZ
					Z	10-9	vsSp	6-3	iPR, xSp	9-5	vsPR, xSp, vsZ
						10-9	vsSp	9-3	vsPR, xSp	9-5	vsPR, xSp, vsZ
						10-9	vsSp	6-3	iPR, xSp	9-5	vsPR, xSp, vsZ
						10-9	vsSp	6-3	iPR, xSp	9-5	vsPR, xSp, vsZ
						10-9	vsSp	6-3	iPR, xSp	9-5	vsPR, xSp, vsZ

16		1.5 W		0.03	X	10–9	vsCrZ	10–9	vsZ	10–9	vsSp, vsZ
						10–9	vsCrZ	10–9	vsZ	10–9	vsSF, vsZ
						10–9	vsCrZ	10–9	vsZ	10–9	vsSp, vsZ
						10–9	vsCrZ	10–9	vsZ	10–9	vsSp, vsZ
						10–9	vsCrZ	10–9	vsZ	10–9	vsSp, vsZ
					Y	10–9	vsCrZ, sEZ	10–9	vsZ	10–9	vsSp, vsZ
						10–9	vsCrZ, sEZ	10–9	vsZ	10–9	vsSp, vsZ
						10–9	vsCrZ, sEZ	10–9	vsZ	10–9	vsSp, vsZ
						10–9	vsCrZ, sEZ	10–9	vsZ	10–9	vsSp, vsZ
						10–9	vsCrZ, sEZ	10–9	vsZ	10–9	vsSp, vsZ
					Z	10–9	vsCrZ, sEZ	10–9	vsZ	9–7	vspR, vsSp, IZ
						10–9	vsCrZ, sEZ	10–9	vsZ	9–7	vspR, vsSp, IZ
						10–9	vsCrZ, sEZ	10–9	vsZ	9–7	vspR, vsSp, IZ
						10–9	vsCrZ, sEZ	10–9	vsZ	9–7	vspR, vsSp, IZ
						10–9	vsCrZ, sEZ	10–9	vsZ	9–7	vspR, vsSp, IZ
17		1.5 W		0.06	X	10–5	xCrE, xsS	9–7	vspR, sCrE	9–7	vspR, sSp, IZ
						10–5	xCrE, xsS	9–7	vspR, sCrE	9–7	vspR, sSp, IZ
						10–5	xCrE, xsS	9–7	vspR, sCrE	9–7	vspR, sSp, IZ
						10–5	xCrE, xsS	8–6	vspR, sCrE	9–7	vspR, sSp, IZ
						10–5	xCrE, xsS	9–7	vspR, sCrE	9–7	vspR, sSp, IZ
					Y	10–8	sCrE	10–8	sCrE	10–8	sSp, IZ
						10–8	sCrE	9–7	vspR, sCrE	10–8	sSp, IZ
						10–8	sCrE	10–8	sCrE	10–8	sSp, IZ
						10–8	sCrE	10–8	sCrE	10–8	sSp, IZ
						10–8	sCrE	10–8	sCrE	10–8	sSp, IZ
					Z	10–5	xCrE	10–8	sCrE	9–7	vspR, sSp, IZ
						10–5	xCrE	10–8	sCrE	9–7	vspR, sSp, IZ
						10–5	xCrE	10–8	sCrE	9–7	vspR, sSp, IZ
						10–5	xCrE	10–8	sCrE	9–7	vspR, sSp, IZ
						10–5	xCrE	10–8	sCrE	9–7	vspR, sSp, IZ

TABLE VI.—*Concluded.*

Lot	Coating, Mils				Plater	Exposure - 1 Year		Exposure - 2 Years		Exposure - 3 Years	
	Strike		BrNi	Cr		Rating	Description	Rating	Description	Rating	Description
18			1.5	0.01 CF	X	9-8	vspR, ssS	8-4	spR, xSp	9-6	vspR, xSp
						9-6	vspR, lsS	7-3	spR, xSp	9-6	vspR, xSp
						10-8	ssS	8-4	spR, xSp	9-6	vspR, xSp
						9-7	vspR, lsS	9-5	spR, xSp	9-6	vspR, xSp
						9-8	vspR, ssS	9-5	spR, xSp	9-6	vspR, xSp
					Y	9-9	vspR, seR	8-5	spR, xSp	8-6	spR, lSp
						9-9	vspR, seR	9-6	spR, xSp	8-6	spR, lSp
						9-9	vspR, seR	8-5	spR, xSp	8-6	spR, lSp
						9-9	vspR, seR	7-4	spR, xSp	8-6	spR, lSp
						9-9	vspR, seR	8-5	spR, xSp	8-6	spR, lSp
19			1.5	0.06 CF$_B$	X	9-9	vspR	9-8	vspR, vsSp	9-8	vspR, sSp
						9-9	vspR	9-8	vspR, vsSp	9-8	vspR, sSp
						9-9	vspR	9-8	vspR, vsSp	9-8	vspR, sSp
						9-9	vspR	8-7	vspR, vsSp	9-8	vspR, sSp
						9-9	vspR	9-8	vspR, vsSp	9-8	vspR, sSp
					Y	10-10		9-9	vspR	9-8	vspR, sSp
						10-10		9-9	vspR	9-8	vspR, sSp
						10-10		9-9	vspR	9-8	vspR, sSp
						10-10		9-9	vspR	9-8	vspR, sSp
20			1.5$_B$	0.01	X	9-9	vspR	9-6	vspR, xSp	7-5	lpR, lSp
						9-9	vspR	9-6	vspR, xSp	7-5	lpR, lSp
						9-9	vspR	7-4	spR, xSp	7-5	lpR, lSp
						7-1	spR, xsS	4-2	xpR, xSp	7-5	lpR, lSp
						7-2	spR, xsS	4-2	xpR, xSp	7-5	lpR, lSp
					Y	10-8	vsSp	9-6	vspR, xSp	9-7	vspR, lSp
						10-8	vsSp	9-6	vspR, xSp	9-7	vspR, lSp
						10-8	vsSp	9-6	vspR, xSp	9-7	vspR, lSp
						10-8	vsSp	9-6	vspR, xSp	9-7	vspR, lSp
						10-8	vsSp	9-6	vspR, xSp	9-7	vspR, lSp

21			0.75	0.01	X	4-4	xPR, xRs	5-3	xPR, xRs, sEZ	6-6	iPR, sEZ
						4-4	xPR, xRs	5-3	xPR, xRs, sEZ	6-6	iPR, sEZ
						4-4	xPR, xRs	5-3	xPR, xRs, sEZ	6-6	iPR, sEZ
						4-4	xPR, xRs	5-3	xPR, xRs, sEZ	6-6	iPR, sEZ
						4-4	xPR, xRs	5-3	xPR, xRs, sEZ	6-6	iPR, sEZ
					Y	3-3	xPR, xRs	3-1	xPR, xRs, sEZ	4-4	xPR, sEZ
						3-3	xPR, xRs	3-1	xPR, xRs, sEZ	4-4	xPR, sEZ
						3-3	xPR, xRs	3-1	xPR, xRs, sEZ	4-4	xPR, sEZ
						3-3	xPR, xRs	3-1	xPR, xRs, sEZ	4-4	xPR, sEZ
						3-3	xPR, xRs	3-1	xPR, xRs, sEZ	4-4	xPR, sEZ
22			2.5	0.01	X	10-8	sSP	8-7	sPR, sSP	9-7	vsPR, iSP, iEZ
						10-8	sSP	8-7	sPR, sSP	9-7	vsPR, iSP, iEZ
						10-8	sSP	8-7	sPR, sSP	9-7	vsPR, iSP, iEZ
						10-8	sSP	8-7	sPR, sSP	9-7	vsPR, iSP, iEZ
						10-8	sSP	8-7	sPR, sSP	9-7	vsPR, iSP, iEZ
					Y	10-7	sSP	8-6	sPR, iSP	9-5	vsPR, xSP
						10-7	sSP	8-6	sPR, iSP	9-5	vsPR, xSP
						10-7	sSP	8-6	sPR, iSP	9-5	vsPR, xSP
						10-7	sSP	8-6	sPR, iSP	9-5	vsPR, xSP
						10-7	sSP	8-6	sPR, iSP	9-5	vsPR, xSP
23			1.5	0.01	X	9-7	vsPR, sSP	6-5	iPR, sSP	9-7	vsPR, iSP
						9-7	vsPR, sSP	6-5	iPR, sSP	9-7	vsPR, iSP
						9-7	vsPR, sSP	6-5	iPR, sSP	9-7	vsPR, iSP
						9-7	vsPR, sSP	6-5	iPR, sSP	9-7	vsPR, iSP
						9-7	vsPR, sSP	6-5	iPR, sSP	9-7	vsPR, iSP
					Y	10-8	sSP, iEZ, sER	8-6	sPR, iSP	9-6	vsPR, xSP
						10-8	sSP, iEZ, sER	8-6	sPR, iSP	9-6	vsPR, xSP
						10-8	sSP, iFZ, sER	8-6	sPR, iSP	9-6	vsPR, xSP
						10-8	sSP, iEZ, sER	8-6	sPR, iSP	9-6	vsPR, xSP
						10-8	sSP, iEZ, sER	8-6	sPR, iSP	9-6	vsPR, xSP

TABLE VII.—INSPECTION DATA, KURE BEACH (800-FT SITE), N. C., EXPOSURE.

ACu = Acid copper, W = Buffed Watts nickel, B = Buffed, CR = Chromium CR 110, CF = Chromium CF 500, CrZ = Chromium crazing, E = Etch. See pages 333 and 334 for other symbols.

Lot	Coating, Mils				Plater	Exposure - 4 Months		Exposure - 7 Months	
	Strike		BrNi	Cr		Rating	Description	Rating	Description
1			1.5	0.01	X	9-9	spR	3-3	lpR, lcR, lRs
						5-5	lpR	3-3	lpR, lcR, lRs
						4-4	xpR	3-3	lpR, lcR, lRs
						9-9	vspR	3-3	lpR, lcR, lRs
						4-4	xpR	3-3	lpR, lcR, lRs
					Y	8-8	spR	6-6	lpR, lRs, vscR
						8-8	spR	6-6	lpR, lRs, vscR
						8-8	spR	6-6	lpR, lRs, vscR
						8-8	spR	6-6	spR, sRs, vscR
						8-8	spR	6-6	spR, sRs, vscR
2	0.05 Cu		1.45	0.01	X	3-3	xpR	2-2	xcR, xRs, xpR
						3-3	xpR	2-2	xcR, xRs, xpR
						4-4	lpR	2-2	xcR, xRs, xpR
						3-3	spR	2-2	xcR, xRs, xpR
						4-4	lpR	2-2	xcR, xRs, xpR
					Y	9-9	vspR	10-10	
						10-10		10-10	
						10-10		10-10	
						8-8	spR, lSp	8-7	vscR, spR, xvsSp
						10-10		10-10	
3	0.05 Ni		1.45	0.01	X	9-9	vspR	4-4	lpR, lcR, lRs
						9-9	vspR	3-3	lpR, lcR, lRs
						9-9	spR	3-3	lpR, lcR, lRs
						9-9	spR	4-4	lpR, lcR, lRs
						9-9	spR	3-3	lpR, lcR, lRs
					Y	9-9	spR	7-6	lpR, scR, sRs, xvsSp
						9-9	spR	7-6	lpR, scR, sRs, xvsSp
						9-9	spR	7-6	lpR, scR, sRs, xvsSp
						8-8	spR, lSp	7-6	lpR, scR, sRs, xvsSp
						9-9	vspR	8-7	spR, vscR, xvsSp
4	0.15 Cu	0.65 ACu	0.7	0.01	X	8-8	spR	6-5	scR, lRs, lpR, xvsSp
						5-5	xpR	5-4	lcR, lRs, lpR, xvsSp
						6-6	lpR	6-5	scR, lRs, lpR, xvsSp
						6-6	lpR	6-5	scR, lRs, lpR, xvsSp
						9-9	vspR	9-8	spR, xvsSp
					Y	10-10		10-9	xvsSp
						10-10		10-9	xvsSp
						10-9	lSp	9-8	xvsSp, vspR
						10-9	lSp	10-9	xvsSp
						10-9	lSp	10-9	xvsSp
5	0.15 Ni	0.65 ACu	0.7	0.01	X	5-5	xpR	3-2	xpR, lRs, xvsSp
						5-5	xpR	3-2	xpR, lRs, xvsSp
						5-5	xpR	3-2	xpR, lRs, xvsSp
						5-5	xpR	3-2	xpR, lRs, xvsSp
						5-5	xpR	3-2	xpR, lRs, xvsSp
					Y	9-9	vspR	7-6	lpR, lRs, vxsSp
						8-8	spR	6-5	lpR, lRs, vxsSp
						9-9	vspR	6-5	lpR, lRs, vxsSp
						9-9	vspR	9-9	vspR
						9-9	vspR	9-9	vspR
6	0.15 Ni		1.35	0.01	X	5-5	xpR	6-5	lpR, lRs, xvsSp
						5-5	xpR	6-5	lpR, lRs, xvsSp
						5-5	xpR	6-5	lpR, lRs, xvsSp
						9-9	vspR	8-7	spR, xvsSp
						4-4	xpR	2-1	xpR, xRs, xvsSp
					Y	5-5	xpR	6-5	lpR, lRs, xvsSp
						5-5	xpR	5-4	lpR, lRs, xvsSp
						9-9	vspR	6-5	lpR, lRs, xvsSp
						9-9	vspR	9-9	vspR
						9-9	vspR	9-9	vspR
7	0.15 Cu		1.35	0.01	X	6-6	lpR	5-5	xcR, xRs, lpR
						6-6	lpR	5-5	xcR, xRs, lpR
						7-7	spR	4-4	xcR, xRs, lpR
						5-5	xpR	4-4	xcR, xRs, lpR
						5-5	xpR	4-4	xcR, xRs, lpR
					Y	9-9	vspR	10-10	
						8-7	spR, lSp	8-7	xvsSp, spR
						10-10		10-10	
						8-7	spR, lSp	7-6	xvsSp, lpR
						8-7	spR, lSp	7-7	lpR

TABLE VII.—*Continued.*

EXPOSURE - 1 YEAR		EXPOSURE - 2 YEARS		EXPOSURE - 3 YEARS	
RATING	DESCRIPTION	RATING	DEACRIPTION	RATING	DESCRIPTION
2-2	xcR, xpR, xRs	1-1	xpR, xRs	0-0	xpR, xRs, icR
3-3	xcR, xpR, xRs	3-3	xcR, xRs	1-0	xpR, xRs, icR
3-3	xcR, xpR, xRs	2-2	xcR, xRs		
2-2	xcR, xpR, xRs	1-1	xpR, xRs	0-0	xpR, xRs, icR
2-2	xcR, xpR, xRs	1-1	xcR, xRs	0-0	xpR, xRs, icR
5-5	iRs, ipR, ieZ	3-2	xpR, xRs, iSp	1-0	xpR, xRs, iSp
5-5	iRs, ipR, ieZ	3-2	xpR, xRs, iSp	1-0	xpR, xRs
5-5	iRs, ipR, ieZ	3-2	xpR, xRs, iSp		
6-6	iRs, ipR, ieZ	4-2	xpR, xRs, iSp	2-0	xpR, xRs
6-6	iRs, ipR, ieZ	2-1	xpR, xRs, iSp	0-0	xpR, xRs
1-1	xcR, xpR, xRs	0-0	xcR, xB	0-0	xcR, xB
1-1	xcR, xpR, xRs	0-0	xcR, xB		
1-1	xcR, xpR, xRs	0-0	xcR, xB	0-0	xcR, xB
1-1	xcR, xpR, xRs	0-0	xcR, xB	0-0	xcR, xB
1-1	xcR, xpR, xRs	0-0	xcR, xB	0-0	xcR, xB
10-10		9-9	vspR	9-9	vspR, sSp
10-10	seZ	10-10			
10-10		10-10		9-8	vspR, sSp
7-7	scR, imB, ipR, iSp	5-3	ipR, xB, iSp		
10-10		10-10		10-9	vspR, sSp
2-2	xpR, xRs	1-1	xpR, xRs	0-0	vxpR, xRs
2-2	xpR, xRs	1-1	xpR, xRs	0-0	vxpR, xRs
2-2	xpR, xRs	1-1	xpR, xRs	0-0	vxpR, xRs
2-2	xpR, xRs	1-1	xpR, xRs		
2-2	xpR, xRs	1-1	xpR, xRs	0-0	vxpR, xRs
5-4	ipR, iRs, isB, imB	1-0	xpR, xcR, xB	0-0	vxB
5-4	ipR, iRs, isB, imB	1-0	xpR, xcR, xB	0-0	vxB
4-3	ipR, iRs, isB, imB	1-1	xpR, sRs, iSp	0-0	vxpR, xRs
4-3	ipR, iRs, isB, imB	2-2	xpR, sRs, iSp	0-0	vxpR, xRs
5-4	ipR, iRs, isB, imB	3-3	xpR, sRs, iSp		
3-3	xcR, iRs, xSp	0-0	vxcR, xRs	0-0	vxcR
2-2	xcR, iRs, xSp	0-0	vxcR, xRs	0-0	vxcR
3-3	xcR, iRs, xSp	0-0	vxcR, xRs	0-0	vxcR
3-3	xcR, iRs, xSp	0-0	vxcR, xRs		
7-5	scR, xSp	3-2	xcR, xRs, iSp	1-0	vxcR
10-10		10-9	sSp	9-8	spR, sSp
10-10		10-10			
9-7	vscR, xSp	9-8	sSp, vspR		
10-8	xSp	10-9	sSp	10-9	sSp
10-8	xSp	10-9	sSp	9-8	spR, sSp
4-3	scR, xpR, xRs, iSp	2-2	xpR, icR, xRs	0-0	xcR, xpR, xRs
4-3	scR, xpR, xRs, iSp	2-2	xpR, icR, xRs	0-0	xcR, xpR, xRs
4-3	scR, xpR, xRs, iSp	2-2	xpR, icR, xRs	0-0	xcR, xpR, xRs
4-3	scR, xpR, xRs, iSp	2-2	xpR, icR, xRs		
4-3	scR, xpR, xRs, iSp	2-2	xpR, icR, xRs	0-0	scR, xpR, xRs
5-4	ipR, scR, xSp	4-2	xcR, ipR, iRs, iSp	1-0	xpR, xcR, xRs, sSp
5-4	ipR, scR, xSp	4-2	xcR, ipR, iRs, iSp		
5-4	ipR, scR, xSp	4-2	xcR, ipR, iRs, iSp	0-0	xpR, xcR, xRs, sSp
9-9	spR	9-8	vspR, sSp	8-7	spR, sSp
9-9	vspR	9-8	vspR, sSp		
4-4	ipR, iRs	3-2	xpR, xRs, sSp, scR	0-0	vxpR, xRs
3-3	xpR, xRs	1-0	xpR, xRs, sSp, scR	0-0	vxpR, xRs
4-4	ipR, iRs	1-0	xpR, xRs, sSp, scR	0-0	vxpR, xRs
6-5	spR, sRs	2-1	xpR, xRs, sSp, scR		
2-2	xpR, xRs	0-0	xpR, xRs, sSp, scR	0-0	vspR, xRs
7-6	iSp, ipR, iRs	7-6	ipR, sRs, sSp	3-2	xpR, xRs
5-4	iSp, ipR, scR	3-2	xpR, iF, iRs, sSp	0-0	xpR, xRs
5-4	iSp, ipR, scR	2-1	xpR, xB, iRs, sSp	0-0	xpR, xRs
8-8	spR	7-6	ipR, sRs, sSp		
8-8	spR	6-5	ipR, sRs, sSp	2-0	xpR, xRs
3-3	xcR, iRs	2-2	vxcR, xB, xRs	0-0	vxB, vxcR
3-3	xcR, iRs	2-2	vxcR, xB, xRs		
2-2	xcR, iRs	0-0	vxcR, xB, xRs	0-0	vxB, vxcR
2-2	xcR, iRs	0-0	vxcR, xB, xRs	0-0	vxB, vxcR
2-2	xcR, iRs	1-1	vxcR, xB, xRs	0-0	vxB, vxcR
10-10		10-9	sSp	9-8	vspR, sSp
8-7	spR, xSp	7-5	ipR, iSp, sRs		
10-10		10-10			
5-4	ipR, xSp	5-3	xpR, iRs, iSp	3-2	xpR, xRs, icR, iSp
4-4	ipR, scR	3-1	xpR, iRs, iSp	0-0	xpR, xRs, icR, iSp

TABLE VII.—*Continued.*

Lot	Coating, Mils				Plater	Exposure - 4 Months		Exposure - 7 Months	
	Strike		BrNi	Cr		Rating	Description	Rating	Description
8			1.5	0.005	X	3-3	xpR	2-2	xpR, xRs
						3-3	xpR	2-2	xpR, xRs
						3-3	xpR	2-2	xpR, xRs
						4-4	xpR	2-2	xpR, xRs
						5-5	xpR	3-3	xpR, xRs
					Y	5-5	xpR	5-5	ipR, sRs
						9-9	vspR	8-8	vspR
						9-9	vspR	9-9	vspR
						9-9	vspR	7-7	spR, sRs
						5-5	xpR	5-5	ipR, sRs
9			1.5	0.03	X	9-9	vspR, xZ	10-9	vsZ
						9-9	vspR, xZ	10-9	vsZ
						10-10	xZ	10-9	vsZ
						9-9	vspR, xZ	10-9	vsZ
						9-9	vspR, xZ	9-8	vsZ, vspR
					Y	7-7	ipR, xZ	9-8	vsZ, vspR
						8-8	spR, xZ	9-8	vsZ, vspR
						9-9	vspR, xZ	9-8	vsZ, vspR
						9-9	vspR, xZ	9-8	vsZ, vspR
						9-9	vspR, xZ	9-8	vsZ, vspR
10			1.5	0.06	X	9-9	vspR, xZ	10-5	vsZ, xE
						10-10	xZ	10-5	vsZ, xE
						10-10	xZ	10-5	vsZ, xE
						10-10	xZ	10-5	vsZ, xE
						10-10	xZ	10-5	vsZ, xE
					Y	9-9	spR, xZ	9-5	vspR, sZ
						8-8	ipR, xZ	9-5	vspR, sZ
						9-9	spR, xZ	9-7	vspR, sZ, vsSp
						9-9	spR, xZ	9-7	vspR, sZ, vsSp
						10-10	xZ	10-5	E
11			1.5	0.005 CR	X	8-8	spR	5-5	ipR, iRs
						9-9	vspR	5-5	ipR, iRs
						9-9	vspR	4-4	ipR, iRs
						9-9	vspR	9-9	spR, sRs
						7-7	xpR	2-2	xpR, xRs
					Y	6-6	ipR, iSp	4-3	ipR, iRs, xvsSp
						5-5	xpR	4-3	ipR, iRs, xvsSp
						6-6	ipR, iSp	4-3	ipR, iRs, xvsSp
						6-6	ipR, iSp	4-3	ipR, iRs, xvsSp
						5-5	xpR	2-2	xpR, xRs
12			1.5	0.01 CR	X	6-4	ipR, xSp	3-2	ipR, iRs, ivsSp
						6-4	ipR, xSp	3-2	ipR, iRs, ivsSp
						9-7	vspR, iSp	6-5	spR, sRs, ivsSp
						5-4	xpR, iSp	2-2	xpR, iRs
						6-4	ipR, iSp	3-2	xpR, iRs, ivsSp
					Y	6-4	ipR, xSp	3-2	xpR, xRs, ivsSp
						6-4	ipR, xSp	3-2	xpR, xRs, ivsSp
						4-4	xpR	3-2	xpR, xRs, ivsSp
						8-4	spR, iSp	4-3	ipR, iRs, ivsSp
						8-4	spR, iSp	6-5	ipR, iRs, ivsSp
13			1.5	0.03 CR	X	9-9	vspR, xZ	9-7	vspR, iZ, sE
						10-10	xZ	10-7	iZ, sE
						9-9	vspR, xZ	9-8	vspR, iZ, sE
						9-9	vspR, xZ	9-8	vspR, iZ, sE
						9-9	vspR, xZ	9-8	vspR, iZ, sE
					Y	9-9	vspR, xZ	9-7	vspR, iZ
						9-9	vspR, xZ	9-7	vspR, iZ
						8-8	spR, xZ	8-6	spR, sRs
						8-8	spR, xZ	9-7	vspR, iZ
						9-9	vspR, xZ	9-7	vspR, iZ
14			1.5	0.06 CR	X	10-10	xZ	10-8	sZ
						9-9	vspR, xZ	10-9	sZ
						9-9	vspR, xZ	9-8	vspR, sZ
						10-10	xZ	10-9	sZ
						10-10	xZ	10-9	sZ
					Y	8-8	spR, xZ	9-8	vspR, sZ
						8-8	spR, xZ	9-7	vspR, sZ
						8-8	spR, xZ	9-8	vspR, sZ
						8-8	spR, xZ	9-8	vspR, sZ
						8-8	spR, xZ	9-8	vspR, sZ

TABLE VII.—*Continued.*

Exposure - 1 Year		Exposure - 2 Years		Exposure - 3 Years	
Rating	Description	Rating	Description	Rating	Description
1-1	xpR, xRs	1-1	vxpR, xRs	0-0	xpR, xRs
1-1	xpR, xRs	1-1	vxpR, xRs	0-0	xpR, xRs
1-1	xpR, xRs	1-1	vxpR, xRs	0-0	xpR, xRs
1-1	xpR, xRs	1-1	vxpR, xRs		
1-1	xpR, xRs	1-1	vxpR, xRs	0-0	xpR, xRs
5-5	ipR, iRs, iSp	2-2	xpR, sSp	0-0	xpR, xRs
7-7	spR, sRs, sSp	5-4	ipR, sSp	2-1	xpR, xRs
9-9	vspR, sSp	8-7	spR, sSp	5-3	ipR, iRs
5-5	ipR, iRs, iSp	2-2	xpR, sSp		
5-5	ipR, iRs, iSp	2-2	xpR, sSp	0-0	xpR, xRs
10-9	crZ	9-8	vspR, iZ	9-8	spR, sE
10-9	crZ	9-8	vspR, iZ	8-7	spR, sE
10-9	crZ	9-8	vspR, iZ	8-7	spR, sE
10-9	crZ	9-8	vspR, iZ	6-5	ipR, sE
10-9	crZ	9-8	vspR, iZ		
8-7	spR, crZ	7-6	spR, iZ		
9-8	vspR, crZ	8-7	spR, iZ	8-6	spR, sE
9-8	vspR, crZ	8-7	spR, iZ	8-6	spR, sE
9-8	vspR, crZ	8-7	spR, iZ	8-6	spR, sE
9-8	vspR, crZ	8-7	spR, iZ	8-6	spR, sE
10-8	sCrE	9-7	spR, xE	9-7	spR, iE
10-8	sCrE	8-6	spR, xE	8-6	spR, iE
10-8	sCrE	9-7	spR, xE	8-6	spR, iE
10-8	sCrE	9-7	spR, xE	9-7	spR, iE
10-8	sCrE	9-7	spR, xE		
9-7	vspR, scrE	8-6	spR, xE	8-6	spR, iE
9-7	vspR, scrE	7-5	spR, xE	7-5	spR, iE
9-7	vspR, scrE	8-6	spR, xE	7-5	spR, iE
9-7	vspR, scrE	8-6	spR, xE	8-6	spR, iE
10-8	scrE	9-7	spR, xE		
2-2	xpR, xRs	1-1	vxpR, xRs, sSp	0-0	vxpR, xRs
2-2	xpR, xRs	1-1	vxpR, xRs, sSp	0-0	vxpR, xRs
2-2	xpR, xRs	1-1	vxpR, xRs, sSp	0-0	vxpR, xRs
6-6	ipR, iRs	2-1	vxpR, xRs, sSp		
0-0	xpR, xRs	1-1	vxpR, xRs, sSp		
2-2	xpR, xRs	1-1	vxpR, xRs, sSp	0-0	vxpR, xRs
3-3	xpR, xRs	2-1	vxpR, xRs, sSp	0-0	vspR, xRs
2-2	xpR, xRs	1-1	vxpR, xRs, sSp		
2-2	xpR, xRs	1-1	vxpR, xRs, sSp	0-0	vspR, xRs
1-1	xpR, xRs	1-1	vxpR, xRs, sSp	0-0	vspR, xRs
1-1	xpR, xRs	1-1	vxpR, xRs, sSp	0-0	vspR, xRs
1-1	xpR, xRs	1-1	vxpR, xRs, sSp	0-0	vxpR, xRs
3-3	xpR, xRs	1-1	vxpR, xRs, sSp	0-0	vxpR, xRs
1-1	xpR, xRs	1-1	vxpR, xRs, sSp	0-0	vxpR, xRs
2-2	xpR, xRs	1-1	vxpR, xRs, sSp		
2-2	xpR, xRs	1-1	vxpR, xRs, sSp	0-0	vxpR, xRs
2-2	xpR, xRs	1-1	vxpR, xRs, sSp	0-0	vxpR, xRs
2-2	xpR, xRs	1-1	vxpR, xRs, sSp		
2-2	xpR, xRs	1-1	vxpR, xRs, sSp	0-0	vxpR, xRs
4-4	ipR, iRs	2-1	vxpR, xRs, sSp	0-0	vxpR, xRs
9-8	vspR, sCrE	9-8	spR, sZ	8-7	spR, sE
10-9	sCrE	9-8	spR, sZ	9-8	spR, sE
9-8	vspR, sCrE	9-8	spR, sZ	9-8	spR, sE
10-9	sCrE	9-8	spR, sZ	9-8	spR, sE
9-8	vspR, sCrE	9-8	spR, sZ		
9-8	vspR, sCrE	9-8	spR, sZ	8-7	spR, sE
9-8	vspR	8-7	spR, sZ	7-6	ipR, sE
8-7	vspR	7-6	spR, sZ		
8-7	vspR, sCrE	7-6	spR, sZ	7-6	ipR, sE
9-8	vspR, sCrE	9-8	spR, sZ	7-6	ipR, sE
10-9	vspR, sCrE	8-7	spR, sZ	6-5	ipR, sE
10-9	vspR, sCrE	8-7	spR, sZ		
9-8	vspR, sCrE	7-6	spR, sZ	6-5	ipR, sE
10-9	vspR, sCrE	8-7	spR, sZ	7-5	ipR, iE
10-9	vspR, sCrE	7-6	spR, sZ	5-3	ipR, iE
9-8	vspR, sCrE	8-7	spR, sZ	8-7	spR, sE
6-5	one v Lb, sCrE	8-7	spR, sZ		
9-8	vspR, sCrE	8-7	spR, sZ	7-5	ipR, iE
9-8	vspR, sCrE	8-7	spR, sZ	6-4	ipR, iE
9-8	vspR, sCrE	7-6	spR, sZ	6-4	ipR, iE

TABLE VII.—*Continued.*

Lot	Coating, Mils				Plater	Exposure - 4 Months		Exposure - 7 Months	
	Strike		BrNi	Cr		Rating	Description	Rating	Description
15		1.5 W		0.01	X	9-9	vspR	9-8	vspR, vsSp
						9-9	vspR	9-8	vspR, vsSp
						9-9	vspR	9-9	vspR,
						10-10		9-9	vspR
						10-10		9-9	vspR
					Y	9-9	vspR	9-9	vspR
						9-9	vspR	9-8	vspR
						9-9	vspR	8-8	vspR
						9-9	vspR	9-9	vspR
						9-9	vspR	9-9	vspR
					Z	9-9	vspR	9-9	vspR
						9-9	vspR	9-9	vspR
						9-9	vspR	9-9	vspR
						9-9	vspR	9-9	vspR
						9-9	vspR	9-9	vspR
16		1.5 W		0.03	X	9-9	vspR, xZ	9-8	vspR, sZ
						9-9	vspR, xZ	9-8	vspR, sZ
						9-9	vspR, xZ	9-8	vspR, sZ
						9-9	vspR, xZ	9-8	vspR, sZ
						9-9	vspR, xZ	9-8	vspR, sZ
					Y	9-9	vspR, xZ	9-8	vspR, sZ
						9-9	vspR, xZ	9-8	vspR, sZ
						9-9	vspR, xZ	9-8	vspR, sZ
						9-9	vspR, xZ	9-8	vspR, sZ
						9-9	vspR, xZ	9-8	vspR, sZ
					Z	9-9	vspR, xZ	9-8	vspR, sZ
						10-10	vspR, xZ	10-9	sZ
						9-9	vspR, xZ	9-8	vspR, sZ
						10-10	vspR, xZ	10-9	sZ
						9-9	vspR, xZ	9-8	vspR, sZ
17		1.5 W		0.06	X	9-9	vspR, xZ	9-6	vspR, sSp, sE
						9-9	vspR, xZ	9-6	vspR, sSp, sE
						9-9	vspR, xZ	9-6	vspR, sSp, sE
						9-9	vspR, xZ	9-6	vspR, sSp, sE
						9-9	vspR, xZ	9-6	vspR, sSp, sE
					Y	9-9	vspR, xZ	9-8	vspR, vsSp, sZ
						9-9	vspR, xZ	9-8	vspR, vsSp, sZ
						9-9	vspR, xZ	9-8	vspR, vsSp, sZ
						9-9	vspR, xZ	9-8	vspR, vsSp, sZ
						9-9	vspR, xZ	9-8	vspR, vsSp, sZ
					Z	10-10	xZ	9-7	vspR, sZ, sE
						9-9	vspR, xZ	9-7	vspR, sZ, sE
						9-9	vspR, xZ	10-8	sZ, sE
						9-9	vspR, xZ	9-7	vspR, sZ, sE
						9-9	vspR, xZ	10-8	sZ, sE
18			1.5	0.01 CF	X	9-9	vspR	9-9	vspR
						9-9	vspR	9-9	vspR
						9-9	vspR	9-9	vspR
						10-10		9-9	vspR
						10-10		9-9	vspR
					Y	7-7	spR	8-8	spR, sRs
						7-7	spR	5-5	xpR, lRs
						7-7	spR	7-7	lpR, lRs
						7-7	spR	8-8	spR, sRs
						7-7	spR	5-5	xpR, lRs
19			1.5	0.06 CFb	X	2-2	xpR, lRs	2-2	xpR, xRs
						2-2	xpR, lRs	2-2	xpR, xRs
						2-2	xpR, lRs	2-2	xpR, xRs
						2-2	xpR, lRs	2-2	xpR, xRs
						2-2	xpR, lRs	2-2	xpR, xRs
					Y	7-7	lpR	5-5	lpR, lRs
						3-3	xpR	2-2	xpR, xRs
						5-5	xpR	2-2	xpR, xRs
						5-5	xpR	2-2	xpR, xRs

TABLE VII.—*Continued.*

Exposure - 1 Year		Exposure - 2 Years		Exposure - 3 Years	
Rating	Description	Rating	Description	Rating	Description
6-6	iPR	5-4	iPR, scR, iRs, sSP		
7-7	iPR	6-5	iPR, iRs, sSP	3-2	xPR, sRs, sSP
6-6	iPR	5-4	iPR, scR, iRs, sSP	3-0	xPR, sRs, sSP, scR
8-8	sPR	6-5	iPR, iRs, sSP	3-2	xPR, sRs, sSP
8-8	sPR	6-5	iPR, iRs, sSP	3-2	xPR, sRs, sSP
8-8	sPR	6-5	iPR, iRs, sSP		
8-8	sPR	6-5	iPR, iRs, sSP	3-2	xPR, xRs, sSP
5-5	iPR	5-4	iPR, iRs, sSP	1-0	xPR, xRs, sSP
8-8	sPR	6-5	iPR, iRs, sSP	2-1	xPR, xRs, sSP
8-8	sPR	6-5	iPR, iRs, sSP	3-2	xPR, xRs, sSP
8-8	sPR	5-4	iPR, iRs, sSP		
8-8	sPR	5-4	iPR, iRs, sSP	2-1	xPR, xRs
8-8	sPR	5-4	iPR, iRs, sSP	1-0	xPR, xRs, scR
9-9	sPR	8-7	iPR, iRs, sSP	4-3	xPR, xRs
9-9	sPR	8-7	iPR, iRs, sSP	6-5	iPR, iRs
6-5	iPR, sCRE	5-4	xPR, sZ		
6-5	iPR, sCRE	5-4	xPR, sZ	3-1	xPR, xRs, sE
6-5	iPR, sCRE	5-4	xPR, sZ	3-1	xPR, xRs, sE
6-5	iPR, sCRE	5-4	xPR, sZ	3-1	xPR, xRs, sE
6-5	iPR, sCRE	5-4	xPR, sZ	3-1	xPR, xRs, sE
9-8	vsPR, sCRE	7-6	sPR, sZ		
8-7	vsPR, sCRE	6-5	sPR, sZ	5-4	iPR, sE
8-7	vsPR, sCRE	7-6	sPR, sZ	5-4	iPR, sE
9-8	vsPR, sCRE	8-7	sPR, sZ	7-6	iPR, sE
9-8	vsPR, sCRE	8-7	sPR, sZ	6-5	iPR, sE
9-8	vsPR, sCRE	8-7	sPR, sZ		
9-8	vsPR, sCRE	9-8	sPR, sZ	9-8	vsPR, sE
9-8	vsPR, sCRE	9-8	sPR, sZ	9-8	vsPR, sE
9-8	vsPR, sCRE	9-8	sPR, sZ	9-8	vsPR, sE
9-8	vsPR, sCRE	9-8	sPR, sZ	9-8	vsPR, sE
8-7	vsPR, sCRE	6-5	iPR, sZ		
9-8	vsPR, sCRE	6-5	iPR, sZ	4-2	iPR, iRs, sE
8-7	vsPR, sCRE	6-5	iPR, sZ	4-2	iPR, iRs, sE
8-7	vsPR, sCRE	6-5	iPR, sZ	4-2	iPR, iRs, sE
8-7	vsPR, sCRE	6-5	iPR, sZ	4-2	iPR, iRs, sE
8-7	vsPR, sCRE	7-6	sPR, sZ		
8-7	vsPR, sCRE	7-6	sPR, sZ	7-6	iPR, iRs, sE
9-8	vsPR, sCRE	8-7	sPR, sZ	7-6	iPR, iRs, sE
8-7	vsPR, sCRE	8-7	sPR, sZ	7-6	iPR, iRs, sE
8-7	vsPR, sCRE	7-6	sPR, sZ	7-6	iPR, iRs, sE
10-9	vsPR, sCRE	9-7	sPR, sZ		
9-8	vsPR, sCRE	7-5	sPR, sZ	6-5	iPR, iRs, sE
10-9	vsPR, sCRE	8-6	sPR, sZ	7-6	iPR, iRs, sE
9-8	vsPR, sCRE	7-5	sPR, sZ	7-6	iPR, iRs, sE
10-9	vsPR, sCRE	9-7	sPR, sZ	9-8	sPR, sRs, sE
4-4	xPR	2-1	sPR, sSP	0-0	xPR, xRs
4-4	xPR	2-1	sPR, sSP		
6-6	iPR	5-4	iPR, sSP	1-0	xPR, xRs
8-8	sPR	7-6	sPR, sSP		
4-4	xPR	4-3	iPR, sSP	1-0	xPR, xRs
6-6	iPR	3-2	xPR, xRs, sSP	0-0	vsPR, vxRs
2-2	xPR	1-0	xPR, xRs, sSP		
6-6	iPR	2-1	xPR, xRs, sSP	0-0	vsPR, vxRs
7-7	iPR	6-5	iPR, xRs, sSP		
2-2	xPR	1-0	xPR, xRs, sSP	0-0	vsPR, vxRs, scR
2-2	xPR				
2-2	xPR	1-1	xPR, icR	0-0	vxPR, xRs
2-2	xPR	1-1	xPR, icR		
2-2	xPR	1-1	xPR, icR	0-0	vxPR, xRs
2-2	xPR	1-1	xPR, icR	0-0	vxPR, xRs
3-3	xPR	4-4	xPR, icR	3-2	xPR, xRs
2-2	xPR	1-1	xPR, icR	0-0	vxPR, xRs
2-2	xPR	1-1	xPR, icR		
2-2	xPR	1-1	xPR, icR	0-0	vxPR, xRs

TABLE VII.—*Continued.*

	Coating, Mils					Exposure - 4 Months		Exposure - 7 Months	
Lot	Strike		BrNi	Cr	Plater	Rating	Description	Rating	Description
20			1.5 B	0.01	X	9-9	vspR	9-9	vspR
						9-9	vspR	9-9	vspR
						10-10		10-10	
						9-9	vspR	9-9	vspR
						9-9	vspR	10-10	
					Y	9-9	vspR	9-9	vspR
						8-8	spR	6-6	lpR, sRs
						9-9	vspR	9-9	vspR
						9-9	vspR	9-9	vspR
						9-9	vspR	9-9	vspR
21			0.75	0.01	X	1-1	xpR, xRs	2-2	xpR, lcR, xRs
						1-1	xpR, xRs	2-2	xpR, lcR, xRs
						1-1	xpR, xRs	2-2	xpR, lcR, xRs
						1-1	xpR, xRs	2-2	xpR, lcR, xRs
						1-1	xpR, xRs	2-2	xpR, lcR, xRs
					Y	9-9	vspR	9-9	vspR
						8-8	spR	8-8	spR
						2-2	xpR, xRs	3-2	lpR, xSp
						2-2	xpR, xRs	1-1	vxpR
						0-0	xpR, xRs	0-0	vxpR
22			2.5	0.01	X	10-9	lSp	10-9	sSp
						9-9	vspR, lSp	8-7	lpR, sSp
						9-9	vspR, lSp	7-6	lpR, sSp
						10-9	lSp	9-8	spR, sSp
						9-9	vspR, lSp	9-8	spR, sSp
					Y	9-9	vspR, lSp	9-9	vspR
						9-9	vspR, lSp	8-7	spR, sSp
						9-9	vspR, lSp	9-9	vspR
						9-9	vspR, lSp	9-9	vspR
						9-9	vspR, lSp	9-9	vspR
23			1.5	0.01	X	4-4	xpR	2-1	xpR, xRs, xSp
						4-4	xpR	2-1	xpR, xRs, xSp
						4-4	xpR	3-2	xpR, xRs, xSp
						4-4	xpR	2-1	xpR, xRs, xSp
						4-4	xpR	2-1	xpR, xRs, xSp
					Y	9-9	vspR	8-8	spR
						9-9	spR	6-6	lpR
						9-9	vspR	7-7	lpR
						9-9	vspR	9-9	vspR
						9-9	vspR	8-8	spR
24		1.5 W		0.05 CFB		6-6	lpR	4-4	xscR, lRs
						8-8	spR	5-4	xscR, lRs
						7-7	spR	4-4	xscR, lRs
						6-6	lpR	4-4	xscR, lRs
						5-5	xpR	4-4	xscR, lRs
25		1.5 W		0.01 CF		9-9	vspR	10-10	
						10-10		10-10	
						9-9	vspR	9-9	spR
						9-9	vspR	9-9	vspR, vscR
						10-10		10-10	
26		1.5 W		0.02		9-9	vspR	9-9	vspR
						10-10		10-10	
						9-9	vspR	9-9	vspR
						10-10		10-10	
						10-10		9-9	vspR

TABLE VII.—*Concluded.*

EXPOSURE - 1 YEAR		EXPOSURE - 2 YEARS		EXPOSURE - 3 YEARS	
RATING	DESCRIPTION	RATING	DESCRIPTION	RATING	DESCRIPTION
9-9	vspR	9-8	spR, sSp		
9-9	vspR	9-8	spR, sSp	9-8	vspR, vsSp
9-9	vspR	10-9	sSp	9-8	vspR, vsSp
9-9	vspR	9-8	spR, sSp	9-8	vspR, vsSp
9-9	vspR	9-8	spR, sSp	9-8	vspR, vsSp
7-7	lpR	5-4	xpR, sSp	4-3	lpR, lRs, lSp
5-5	lpR	3-2	xpR, sSp		
9-9	vspR	9-8	spR, sSp		
9-9	vspR	8-7	spR, sSp	6-5	lpR, lRs, lSp
9-9	vspR	8-7	spR, sSp	6-5	lpR, lRs, lSp
1-1	xpR	0-0	vxpR, xcR		
1-1	xpR	0-0	vxpR, xcR	0-0	vxcR, xRs
1-1	xpR	0-0	vxpR, xcR	0-0	vxcR, xRs
1-1	xpR	0-0	vxpR, xcR	0-0	vxcR, xRs
1-1	xpR	0-0	vxpR, xcR	0-0	vxcR, xRs
9-9	vspR	7-6	lpR, sSp		
7-7	spR	4-3	xpR, sSp	1-0	vxpR, xRs
0-0	xpR	0-0	xpR, lcR, sSp	0-0	vxpR, xRs
0-0	xpR	0-0	xpR, lcR, sSp	0-0	vxpR, xRs
0-0	xpR	0-0	xpR, lcR, sSp		
6-6	lpR	4-3	xpR, sSp		
5-5	lpR	3-2	xpR, sSp	1-0	vxpR, xRs
3-3	xpR	3-2	xpR, sSp	0-0	vxpR, xRs
6-6	lpR	4-3	xpR, sSp	2-0	xpR, xRs
5-5	lpR	4-3	xpR, sSp	2-0	xpR, xRs
9-9	vspR	9-8	spR, sSp	8-7	spR, sRs, sSp
7-7	spR	6-5	lpR, sSp	5-3	lpR, lRs, sSp
8-8	spR	6-5	lpR, sSp	6-5	lpR, sRs, sSp
7-7	spR	6-5	lpR, sSp		
5-5	lpR	4-3	spR, sSp	2-1	xpR, lRs, lSp
0-0	xpR	0-0	vxpR		
0-0	xpR	0-0	vxpR	0-0	xcR, xpR, xRs
0-0	xpR	0-0	vxpR	0-0	xcR, xpR, xRs
0-0	xpR	0-0	vxpR	0-0	xcR, xpR, xRs
0-0	xpR	0-0	vxpR	0-0	xcR, xpR, xRs
6-6	lpR	4-3	xpR, sSp	1-0	xpR, xRs
3-3	xpR	1-0	xpR, sSp	1-0	xpR, xRs
3-3	xpR	1-0	xpR, sSp		
7-7	spR	5-4	xpR, sSp	0-0	xpR, xRs
7-7	spR	4-3	xpR, sSp		
2-2	xpR	1-1	vxcR, xB, xRs	0-0	vxcR, xB, xRs
3-3	xpR	2-2	vxcR, xB, xRs	0-0	vxcR, xB, xRs
2-2	xpR	1-1	vxcR, xB, xRs	0-0	vxcR, xB, xRs
2-2	xpR	1-1	vxcR, xB, xRs		
2-2	xpR	0-0	vxcR, xB, xRs	0-0	vxcR, xB, xRs
9-9	vspR	8-8	spR	5-4	sSp, lpR, scR, sRs, seB, seR
10-10		9-9	spR	8-7	spR, sSp
9-9	vspR	9-9	spR		
8-8	spR	7-7	spR, sB	4-2	lpR, lB, lRs, sSp
10-10		10-10		9-8	vspR, sSp, lepR
9-9	vspR	8-7	spR, sSp	7-6	lpR, sRs, sSp
9-9	vspR	8-7	spR, sSp		
9-9	vspR	8-7	spR, sSp	7-6	lpR, sRs, sSp
9-9	vspR	9-8	spR, sSp	8-7	spR, sRs, sSp
9-9	vspR	8-7	spR, sSp	7-6	lpR, sRs, sSp

TABLE VIII.—COMPARISON OF RATING WITH COATING THICKNESS.

Exposure: 2-yr Exposure at Kure Beach, N.C.

Lot No.	Plater	Measured Thickness, mils				Rating Numbers		Description
		Strike	Copper	Nickel	Chromium	Protection	Appearance	
No. 1	*X*	...	...	1.35	0.008	2	2	xcR, xRs
	Y	...	...	1.79	0.009	3	2	xpR, xRs, iSp
No. 2	*X*	Copper 0.05	...	1.38	0.010	0	0	xcR, xB (broken)
	Y	Copper 0.05	...	2.05	0.004	10	10	...
	Y	Copper 0.05	...	1.97	0.004	5	3	ipR, xB, iSp
No. 3	*X*	Nickel 0.05	...	1.15	0.010	1	1	xpR, xRs
	Y	Nickel 0.05	...	1.45	0.008	3	3	xpR, xRs, iSp
No. 4	*X*	Copper 0.85	...	0.76	0.008	0	0	vxcR, xRs
	Y	Copper 0.98	...	0.96	0.008	10	10	...
	Y	Copper 0.99	...	0.69	0.007	9	8	sSp, vspR
No. 5	*X*	Nickel 0.13	0.56	0.69	0.010	2	2	xpR, icR, xRs
	Y	Nickel 0.14	0.57	0.66	0.005	4	2	xcR, ipR, iRs, iSp
	Y	Nickel 0.14	0.30	0.73	0.007	9	8	vspR, sSp
No. 6	*X*	Nickel 0.14	...	1.31	0.009	2	1	xpR, xRs, sSp, scR
	Y	Nickel 0.13	...	1.69	0.007	7	6	ipR, sRs, sSp
No. 7	*X*	Copper 0.16	...	1.35	0.010	2	2	vxcR, xB, xRs
	Y	Copper 0.12	...	1.26	0.009	10	10	...
	Y	Copper 0.13	...	1.82	0.005	3	1	xpR, iRs, iSp
No. 8	*X*	...	...	1.57	0.009	1	1	vxpR, xRs
	Y	...	...	2.00	0.003	2	2	xpR, sSp
No. 9	*X*	...	...	1.54	0.048	9	8	vspR, iZ
	Y	...	...	2.09	0.038	7	6	spR, iZ
No. 10	*X*	...	...	1.50	0.093	9	7	spR, x etch
	Y	...	...	2.08	0.084	9	7	spR, x etch
No. 11	*X*	...	...	1.53	0.005	2	1	vxpR, xRs, sSp
	X	...	...	1.44	0.005	1	1	vxpR, xRs, sSp
	Y	...	...	1.91	0.005	1	1	vxpR, xRs, sSp
No. 12	*X*	...	...	1.47	0.010	1	1	vxpR, xRs, sSp
	Y	...	...	1.82	0.010	1	1	vxpR, xRs, sSp
No. 13	*X*	...	...	1.51	0.030	9	8	spR, sZ
	Y	...	...	1.69	0.030	9	8	spR, sZ
No. 14	*X*	...	...	1.39	0.070	8	7	spR, sZ
	Y	...	...	2.15	0.060	8	7	spR, sZ
No. 15	*X*	...	...	1.63	0.018	5	4	ipR, scR, iRs, sSp
	Y	...	...	1.82	0.012	6	5	ipR, iRs, sSp
	Z	...	...	1.64	0.018	5	4	ipR, iRs, sSp
No. 16	*X*	...	...	1.47	0.054	5	4	xpR, sZ (surface)
	Y	...	...	1.64	0.037	7	6	spR, sZ
	Z	...	...	1.81	0.042	8	7	spR, sZ
No. 17	*X*	...	...	1.86	0.105	6	5	ipR, sZ (surface)
	Y	...	...	1.75	0.070	7	6	spR, sZ
	Z	...	...	1.83	0.096	9	7	spR, sZ
No. 18	*X*	...	...	1.50	0.010	2	1	xpR, sSp
	X	...	...	1.51	0.009	7	6	spR, sSp
	Y	...	...	1.47	0.010	1	0	xpR, xRs, sSp
	Y	...	...	1.81	0.010	6	5	ipR, xRs, sSp
No. 19	*X*	...	...	1.64	0.065	1	1	xpR, icR
	Y	...	...	2.84	0.058	1	1	xpR, icR
No. 20	*X*	...	...	1.59	0.007	9	8	spR, sSp
	Y	...	...	2.12	0.012	3	2	xpR, sSp
	Y	...	...	2.05	0.009	9	8	spR, sSp
No. 21	*X*	...	...	0.73	0.010	0	0	vxpR, xcR
	Y	...	...	0.49	0.010	7	6	ipR, sSp
	Y	...	...	0.50	0.005	0	0	xpR, icR, sSp
No. 22	*X*	...	...	2.52	0.011	4	3	xpR, sSp
	Y	...	...	3.14	0.007	6	5	ipR, sSp
No. 23	*X*	...	...	1.42	0.008	0	0	vxpR
	Y	...	...	1.53	0.006	1	0	xpR, sSp
	Y	...	...	2.06	0.009	4	3	xpR, sSp
No. 24	*Z*	...	...	1.69	0.048	1	1	vxcR, xB, xRs
No. 25	*Z*	...	...	1.81	0.009	9	9	xpR
No. 26	*Z*	...	...	1.71	0.016	8	7	spR, sSp

APPENDIX II

EXPOSURE AND ACCELERATED TESTS OF SUPPLEMENTARY CHROMATE TREATMENTS FOR ELECTROPLATED ZINC COATINGS[1]

REPORT OF SECTION B OF SUBCOMMITTEE V ON SUPPLEMENTARY TREATMENT FOR METALLIC COATINGS

This final report summarizes the results of an investigation of chromate conversion films for electroplated zinc coatings on steel. Its purpose was to explore the possibility of correlating service performance with one or more accelerated testing procedures.

There are more than two dozen varieties of chromate treatments for zinc available on the market. With few exceptions, these treatments are either proprietary solutions or are operated under license. For the sake of simplicity, these treatments can be grouped into four major classifications: bright, clear finishes which are primarily used for appearance; bronze-colored iridescent finishes which are generally used for protection and where appearance is of secondary importance; dark colored treatments for heavy duty that may also be used as a base for organic finishes; and treatments applied by electrochemical means.

Chromate Treatments:

The following typical chromate treatments were used in this investigation: (*a*) an electrolytic treatment of the clear type; (*b*) a yellow electrolytic treatment; (*c*) a plain-dip olive-drab-colored film representing the dark variety; (*d*) a clear dip treatment; (*e*) two iridescent dip treatments used primarily for corrosion protection: one a proprietary treatment coded with suffix 1; and one of known composition representing an earlier type of chromate treatment for zinc coded with suffix 2. All these treatments, with the exception of the last one, are proprietary solutions. Treatment (*f*) is applied from a 20 per cent sodium dichromate solution acidified with sulfuric acid to a pH of around 1.0.

Preparation Procedures:

For this study, the treatments were applied to nominal 0.0005 - in. thick electroplated zinc coatings on cold-rolled steel panels. The plating was carried out in a conventional manner as follows:

1. Degreased in trichloroethylene,
2. Anodically cleaned in a hot proprietary alkaline cleaner,
3. Spray rinsed,
4. Dipped in 20 per cent by volume hydrochloric acid,
5. Spray rinsed, and
6. Electroplated.

The zinc cyanide plating bath used was purified by treatment with zinc dust and sodium sulfide, and by filtration through activated carbon. No agitation was used. The bath composition and operating conditions are:

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[1] The outdoor exposure tests were conducted by Subcommittee II on Performance Tests, and identified as Program No. 101.

Zinc cyanide	66 g per liter
Sodium cyanide	31 g per liter
Sodium hydroxide	54 g per liter
Temperature	Room (85 to 95 F)
Current density	14 amp per sq ft
Time[a]	34 to 41 min

[a] Depending on thickness checks on specimen panels and on varying allowance for thickness of zinc removed by chromate treatment.

The thickness of zinc coating was held to within ±10 per cent of the nominal, with a few exceptions as noted below.

Variations over a given panel are significantly larger in the case of the yellow-electrolytic and the clear-dip treatments.

TABLE I.—AVERAGE THICKNESS OF SAMPLE PANELS.

Treatment	Thickness, in.
Clear electrolytic	47, 49, 48, 49, 50 48 $\times 10^{-5}$
Yellow electrolytic	47, 48, 46, 45, 54, 48 $\times 10^{-5}$
Olive drab	50, 51, 51, 48, 47 $\times 10^{-5}$
Clear dip	42, 57, 49, 45, 51 $\times 10^{-5}$
Iridescent-1	42, 42, 47, 46, 43, 42 $\times 10^{-5}$
Iridescent-2	49, 44, 53 $\times 10^{-5}$
Untreated zinc	54, 53, 51 $\times 10^{-5}$.

The average thickness of the iridescent-1 treated panels is slightly lower, and that of the untreated zinc somewhat higher than nominal.

A résumé of the average thickness readings for several panels of each treatment is shown in Table I. Thickness measurements were made with a Magne-Gage at several points over the entire panel surface to within ½ in. from the edges.

The supplementary chromate treatments were applied immediately after plating, in accordance with the instructions in the manufacturer's bulletins. The actual application of each treatment was supervised by a representative of the manufacturer. The finished and properly rinsed panels were dried quickly with a blast of cold filtered air and were stored in a relatively clean office atmosphere for 24 hr. They were then placed in polyethylene bags and stored until ready for test. For purposes of comparison, unchromated zinc panels were included in these tests. The panels were kept in storage in the polyethylene bags for about 10 to 11 months before any testing was begun.

Exposure Tests:

The variously treated and untreated zinc-plated panels, in triplicate, were exposed to the following tests:

Atmospheric Exposures.—Sets of test specimens were exposed to the following atmospheric environments:

New York, N. Y.—An industrial atmosphere with some saline contamination located on the roof of the Bell Telephone Laboratories.

Kure Beach, N. C.—A marine location, about 800 ft from the beach line.

These were bold exposures, with the panels inclined 30 deg from the horizontal, facing south.

Sheltered Exposures.—One set was placed in an almost upright position in an unheated building at the Kure Beach location. This was intended to represent exposure to an unheated warehouse atmosphere. Another set was stored within a closed wooden packing box and placed in the same building with the view of checking the effect of this type of exposure.

Accelerated Tests:

The exposures described above were designed to yield information on typical service behavior. Treated panels were also subjected to the following accelerated testing procedures:

- Salt Spray
- High Humidity with cycling temperature
- Total Immersion—continuous (in running tap water)
- Total Immersion—static (in aerated distilled water)
- Hexavalent Chromium Ion Analysis
- Various Spot Tests

A description of these tests follows:

Salt Spray Test.—This test was conducted in accordance with ASTM Method B 117 - 49 T[2] employing a 20 per cent salt solution. The chromated panels were scored with two diagonal scratches in an attempt to evaluate the inhibiting properties of these treatments.

Humidity Test.—This test consists of exposure to a relative humidity of 90 per cent or more while the temperature is being cycled between 80 and 120 F twice daily. The specimens are wet with condensed atmospheric moisture during a considerable part of the rise-in-temperature period. The panels in this test were also scored as above.

Total Immersion—Continuous.—This test consisted in immersing the panels in running tap water for one week. The water temperature was about 62 F.

Total Immersion—Static.—One set of test panels was immersed for one week in continually aerated distilled water maintained at 100 F. The water was changed once after the first few hours on the assumption that most of the soluble chromate had been leached out. For the remainder of the time, the same water was used.

Hexavalent Chromium Ion Analysis.—In this test the amount of soluble Cr^{6+} was determined quantitatively by a standard diphenylcarbazide comparative test method.

Spot Tests.—Several spot tests were investigated. This testing procedure is based on the dissolving action of the testing solution on the film. A color change denotes the end point. In performing this test, a drop of a test solution is applied to the coated surface. With certain solutions the drop is allowed to remain for a fixed time after which any color change is noted. With others, a change in color is taken as a criterion of film quality. Three solutions were tested:

SOLUTION A.—A mixture of 0.75 g basic copper carbonate (green) and 120 g ammonium chloride per liter, adjusted to a pH of 5.8. A drop of solution is allowed to remain on the surface for 30 sec. The appearance of a black or dark gray spot is indicative of an inadequate chromate film. This test is usually employed to establish the presence or absence of a coating and does not necessarily differentiate between films of varying qualities.

SOLUTION B.—This proprietary solution consists of two separate chemicals which are mixed in equal volumes just prior to use. The color of the drop will change from a light amber to blue-green. The time required for this color to develop varies with the thickness of coating and with its quality.

SOLUTION C.—This is 3 per cent sodium chloride solution containing a dye (bromphenol blue) which changes color as the result of changes in pH due to zinc corrosion. The pH of the solution is adjusted to the exact color change-over point (just yellow). The time to color change of the drop is recorded.

TEST RESULTS

The results from these various exposures and accelerated tests are as follows:

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[2] Tentative Method of Salt Spray (Fog) Testing (B 117 - 49 T), 1949 Book of ASTM Standards, Part 2, p. 786.

Outdoor Exposure—New York, N. Y.

Coating	Per Cent Red Rust[a],[b]			
	24 months	26 months	29 months	36 months
Clear electrolytic	5	15	65	100
Yellow electrolytic	5	70	88	100
Olive drab	10	80	96	100
Clear—plain dip	8	35	67	100
Iridescent-1	10	70	77	100
Iridescent-2	5	30	30	100
Untreated zinc	8	35	42	100

[a] These values are averages of 3 panels for the surfaces facing skyward. The groundward surfaces had about 10 per cent red rust at the conclusion of the test after 36 months.

[b] No white corrosion developed during this outdoor exposure.

Outdoor Exposure—Kure Beach (800-ft site), N. C.

Coating	Per Cent White Corrosion[a]			
	21 months		34 months[b]	46 months[c]
	Front	Back		
Clear electrolytic	80	25	>50	light film, no rust
Yellow electrolytic	50	8		
Olive drab	60	10		
Clear—plain dip	15	15		
Iridescent-1	40	6		
Iridescent-2	65	7		
Untreated zinc	85	65		

[a] These values are averages of 3 panels.

[b] No significant difference in white corrosion products on either treated or untreated zinc on skyward side. Light amount of rusting on some panels beginning to appear. On groundward side slight protection against white corrosion by treatments noticeable, but not significantly different.

[c] Appearance about same after 58 months exposure.

Sheltered Exposure—Kure Beach (800-ft site), N. C.

Exposed 21 Months.—No white corrosion products. Some fading of treatment colors, probably due to dust and moisture accumulation.

Exposed 34 Months.—No significant increase in corrosion. No clear cut indication of superiority of one treatment over any other from the standpoint of preventing white corrosion. Untreated zinc appeared to be least corroded.

Exposed 46 and 58 Months.—Changes from previous inspections very slight.

Packaged Exposure in Sheltered Area at Kure Beach

Exposed 21 Months.—Less than 1 per cent of total area of any one panel corroded in the form of small white spots. No significant difference among treated and untreated zinc.

Exposed 34 Months.—All panels show minor amount of white corrosion spots, some with definite red rust centers. There is no significant difference among any treated and untreated zinc.

Exposed 46 Months.—Deterioration of all specimens slightly more advanced, but still no noticeable difference among treatments.

Exposed 58 Months.—Changes from previous inspection very slight.

Salt Spray Test per ASTM Specification B 117 - 49 T

Observations were made after 24 hr and again after 96 hr exposure.

Exposed 24 Hours:

Clear-Electrolytic Treatment showed few white corrosion spots in scratches. The remaining specimens were satisfactory (scattered black spots).

Yellow-Electrolytic Treatment satisfactory (slight color fading).

Olive Drab Treatment showed few white corrosion spots in scratches and a few scattered on rest of area.

Clear Dip Treatment showed few white corrosion spots in scratches and on rest of area.

Iridescent-1 Treatment showed very few small white corrosion spots in scratches; scattered white corrosion spots on rest of area.

Iridescent-2 Treatment showed few small white corrosion spots in scratches; remainder satisfactory.

Untreated Zinc resulted in 100 per cent white corrosion; no rust.

Exposed 96 hr:

Treatment	Per Cent White Corrosion	Red Rust
Clear electrolytic	95	very slight
Yellow electrolytic	15	none
Olive drab	<5 (mostly in scratch)	none
Clear dip	50	none
Iridescent-1	15	none
Iridescent-2	<10	none
Untreated zinc	100	some

Treatments which showed a small amount of white corrosion, namely yellow-electrolytic, olive drab, iridescent-1, and iridescent-2, offered considerable protection in the scratch areas.

HUMIDITY TEST

Exposed 20 Months.—Very little white corrosion in form of small spots on any of the treated specimens. Even the untreated zinc is practically uncorroded. The test was discontinued.

HEXAVALENT CHROMIUM ION ANALYSIS

Treatments	Cr^{6+}, μg per sq in.
Clear electrolytic	2.3
Yellow electrolytic	100
Olive drab	54
Clear dip	0.5
Iridescent-1	87
Iridescent-2	56

SPOT TESTS

Solution A:

All treatments showed presence of film.

Solution B:

Treatments	Time, min, to Blue-Green Color, Two Determinations
Clear electrolytic	2,1.5
Yellow electrolytic	2,1
Olive drab	11,6
Clear dip	3,3
Iridescent-1	4,3
Iridescent-2	8,5
Untreated zinc	0.25,...

Solution C:

In this test color began to change at outer edge of the drop and the time given is for complete change.

Treatments	Time, min, for Complete Color Change—Two Determinations
Clear electrolytic	2.0, 6.5
Yellow electrolytic	5.5, 8.75
Olive drab	4.5, 7.75
Clear dip	8.0, 7.75
Iridescent-1	4.5, 10.0
Iridescent-2	10.0, 13.0
Untreated zinc	>10, 8.5

IMMERSION

Continuous Immersion:

Duration of Test.—One week.

Observations.—Corrosion very light and differences very small; test inconclusive.

Static Immersion:

Duration of Test.—One week.

Observation.—Very little corrosion and very small differences; test inconclusive.

DISCUSSION AND CONCLUSIONS

The results from these tests clearly show that the intent which prompted this investigation was not realized. Correlation between service performance and accelerated tests could not be established. Most accelerated tests yielded inconclusive results. Only the salt spray test and a spot test (solution B) indicated some performance difference, as did the hexavalent chromium ion test. However, the data obtained from the various service-type exposures failed to reveal any significant difference among the six treatments.

An unexpected behavior was that of untreated zinc. Its performance was equal and in some cases superior to that of treated zinc. This was particularly noticeable in the humidity and packaging tests, where untreated zinc normally fails by white corrosion considerably sooner than does chromate treated zinc.

The fact that all specimens used in this investigation had been stored for a long period (10 months) in polyethylene bags may be the basis for a possible explanation of these unexpected behaviors. The untreated zinc coatings developed a light gray coating during the storage period in the polyethylene bags. It is possible that this may have been caused by moisture entrapped in the polyethylene. Failure of the untreated zinc to develop white corrosion in the humidity test may be due to this incipient corrosion film which acted as a barrier to further attack. It is well known that a freshly prepared zinc coating will white-corrode within one to two cycles in this humidity test. No deleterious effect was noted on the panels tested.

Other factors which may have contributed to producing these inconclusive results are: the storage period between preparation and testing was excessive; the sheltered area used was not truly representative of severe storage conditions; the storage box was made of a well-seasoned wood that is normally not used for this purpose. It is recommended that these factors be carefully considered in any further evaluations of this kind.

Respectfully submitted on behalf of the subcommittee,

A. MENDIZZA,
Chairman.

REPORT OF COMMITTEE B-9
ON
METAL POWDERS AND METAL POWDER PRODUCTS*

Committee B-9 on Metal Powders and Metal Powder Products held one meeting during the year: in St. Louis, Mo., on Feb. 27, 1961.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods and specification as appended hereto:[1]

Tentative Method of Test for:

Density of Sintered Metal Friction Materials, and

Determination of Transverse Rupture Strength of Sintered Metal Friction Materials.

Tentative Specifications for:

Steel-Backed Metal Powder Bearing and Bushing Alloys.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Specifications for Copper-Infiltrated Sintered Metal Powder Structural Parts (B 303 - 58 T):[2]

Title.—Change the title to read "Specifications for Copper-Infiltrated Sintered Carbon Steel Structural Parts."

Table I.—Delete the chemical requirements for silicon and aluminum, replacing these with: "Acid insolubles, max, per cent .... 0.5."

Change the values of Combined carbon (on basis of iron only), per cent, for Class B to read: "0.26 to 0.60" and for Class C to read: "0.61 to 1.00."

Section 5.—Delete Paragraphs (*d*) and (*e*) pertaining to the method for density determination, since this test method is covered by Method B 328.

Tentative Method of Test for Compressibility of Metal Powders (B 331 - 58 T):[3]

Section 4(*b*).—Add the following note at the end of this paragraph:

NOTE.—If the powder is to be used in the production of high-density parts, a suitable pressure up to 120,000 psi may be used.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Method of Test for Apparent Density of Refractory Metals and Compounds by the Scott Volumeter (**B 329 - 58 T**)[3] be approved for reference to letter ballot of the Society for adoption as standard without revision.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

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* Sixty-fourth Annual Meeting of the Society, June 25-30, 1961.

[1] The new tentative methods appear in the 1961 Book of ASTM Standards, Part 3, and the new tentative specification appears in the 1961 Book of ASTM Standards, Part 2.

[2] 1958 Book of ASTM Standards, Parts 1 and 2.

[3] 1958 Book of ASTM Standards, Part 3.

Standard Specifications for Iron Copper Sintered Metal Powder Structural Parts (B 222 – 58):[2]

Section 4.—Delete the chemical requirements for silicon and aluminum, replacing these with: "Acid insolubles, max, per cent .... 0.5." Change the requirement for Total other elements determined by difference, max, per cent, from "2.5" to "1.5."

Section 5.—Delete Paragraphs (*d*) and (*e*) pertaining to the method for obtaining density, since it is covered in Method B 328.

Standard Definitions of Terms Used in Powder Metallurgy (B 243 – 58):[4]

New Definition.—Add the following new definition:

Sinterings.—A product made by sintering metal powders. Synonymous with Powder Metallurgy Parts, and Sintered Metal Powder Parts.

Standard Specifications for Bronze Sintered Metal Powder Structural Parts (B 255 – 60):[4]

Section 5(a).—Change "Class A" to read "Type I" and change the density tolerance to read "6.40 to 6.80." Change "Class B" to read "Type II" with a density tolerance of "6.81 to 7.20."

Delete Paragraphs (*d*) and (*e*) pertaining to the method of obtaining density, since it is covered in Methods B 328.

STANDARDS CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following standards:

Standard Methods of Test for:

Apparent Density of Metal Powders (**B 212 – 48**),

Flow Rate of Metal Powders (**B 213 – 48**), and

Density and Interconnected Porosity of Sintered Powder Metal Structural Parts and Oil-Impregnated Bearings (**B 328 – 60**).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Methods of Test for:

Green Strength of Compacted Metal Powder Specimens (**B 312 – 58 T**), and

Average Particle Size of Refractory Metals and Compounds by Fisher Subsieve Sizer (**B 330 – 58 T**).

Tentative Specification for:

Metal Powder Sintered Bearings (Oil Impregnated) (**B 202 – 60 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ADVISORY COMMITTEE ACTIVITIES

The Advisory Committee held two meetings during the year. Committee personnel changes were reviewed and approved. Roland P. Koehring, Chairman of Section III, a Charter Member of Committee B-9, and extremely active in the work of the committee, resigned from Committee B-9 and from the Chairmanship of Section III. His coming retirement from business precipitated his resignation. H. R. Biehl was appointed as the new Chairman of Section III. He will continue as Vice-Chairman of Committee B-9. J. I. Farmer was appointed Chairman of Subcommittee III, Section A, on Bearings to replace M. H. Meighan who resigned. The activities of the subcommittees were reviewed, and topics for extending the work of several committees were studied.

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[4] 1958 Book of ASTM Standards, Part 2.

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

ACTIVITIES OF SUBCOMMITTEES

The seven active subcommittees and sections met twice during the year: in ASTM Headquarters, on Oct. 17 and 18, 1960, and prior to the main committee meeting in St. Louis, Mo. on Feb. 27 and 28, 1961. Subcommittee III, Section D on Friction Materials met an additional time on May 17, 1960, in Cleveland, Ohio.

Subcommittee I on Nomenclature and Technical Data (F. N. Rhines, chairman) obtained approval of the term "Sinterings" to be added to the glossary of terms used in powder metallurgy, thus ending 17 years of effort to adopt a term describing the subject product of Committee B-9. A paper written by E. Geijer, entitled "Effect of Different Furnaces and Sintering Practices on the Strength of Sintered Iron Powder" was accepted for publication by ASTM in *Materials Research & Standards.* Frank Emley, heading a task group, is investigating the need for revision of the standard flat tension test bar. P. V. Schneider is conducting a survey of the need for a standard impact bar and testing procedure for sinterings. It is felt that such a test will become increasingly important as sinterings of high density become more common.

Subcommittee II on Metal Powders (J. B. Haertlein, chairman):

Section A on Base Metal Powders (J. B. Haertlein, chairman).—S. L. Forss conducted a cooperative test program on the compressibility of metal powders at high densities, and found excellent agreement could be obtained using the procedures of Method B 331. As a result a note was added to Method B 331 as referred to earlier in this report. The data and report on the effect of furnace conditions on the sintering of a copper-tin mix has not been published as yet because of the expense of the desired colored photomicrographs. Interest in a test method for sieve analysis and for apparent density of nonfree-flowing metal powders has reached the discussion stage. The committee recommended the continuation of Methods B 212 and B 213 as standard without change and Method B 312 as a tentative without change as reported earlier in this report.

Section B on Refractory Metal Powders (W. H. Bleecker, chairman) reports that results of an interlaboratory test on turbidimetric analysis indicated a much closer agreement than previously experienced because the method of deagglomerating the powder prior to analysis was specified in detail. The data are being distributed so that a narrowing of methods and medium for de-agglomerating can be performed for another interlaboratory test. Unsuccessful attempts were made to prepare permanent slides for an interlaboratory test on microscopic count procedure. As a result of the October meeting, a report was written summarizing the work to date so that further course of action could be decided. The committee recommended the microscopic count method of test be considered unsatisfactory, as a possible standard for refractory metal powders, and that a footnote be added to Recommended Practice E 20 to the effect that this practice is not recommended as a standard for refractory metal powders by Subcommittee II-B of Committee B-9. The committee recommended the adoption as standard of Method B 329 and the continuation of Method B 330 as tentative as reported earlier in this report.

Subcommittee III on Metal Powder Products (R. P. Koehring, chairman):

Section A on Bearings (J. I. Farmer, chairman) submitted to ASTM for publication the paper correlating machinability and microstructure of sintered copper-tin bronze. The committee recommended the proposed Tentative Specifications for Steel-Backed Metal Powder Bearing and Bushing Alloys referred to earlier in this report as well as the continuation of Method B 202 as tentative and Method B 328 as standard. A new task force has been organized to prepare specifications for a copper-tin steel backed bearing impregnated with approximately 50 per cent lead.

Section B on Structural Parts (P. J. Shipe, chairman) took action to include in Specifications B 310 a sintered steel, having a density above 6.5 g per cu cm. A letter ballot proved the results were not acceptable and resulted in changes that will be ballotted upon in the committee. A task force is preparing a specification of iron-copper carbon sintered alloys. A task force studying chemical tolerances of insolubles in Specification B 282 reported that the limits are satisfactory. The committee recommended revisions of Specifications B 222, B 303, and B 255 as indicated earlier in this report. The committee is considering the inclusion of impact and fatigue strength data in the appendix of specifications.

Section C on Cemented Carbides (O. W. Reen, chairman) voted by letter ballot upon the following procedures prepared by the Cemented Carbide Producers Association:

1. Tentative Procedure for the Determination of Transverse Rupture Strength of Cemented Carbides,
2. Tentative Procedure for the Determination of Compressive Strength of Cemented Carbides,
3. Tentative Procedure for the Determination of Poisson's Ration of Cemented Carbides,
4. Tentative Procedure for the Determination of Young's Modulus of Cemented Carbides,
5. Tentative Procedure for the Determination of Electrical Resistivity of Cemented Tungsten Carbides, and
6. Tentative Procedure for the Determination of Linear Thermal Expansion of Cemented Carbides.

The Procedure for Determining the Transverse Rupture Strength of Cemented Carbides was adopted by letter ballot within the committee without revision. The balance of the procedures were not adopted because of the repetition of current ASTM procedures in the text. Mr. John Redmond was appointed to revise the procedures in such a form that they could be incorporated into existing ASTM standard test procedures now under the jurisdiction of Committees E-1 on Methods of Testing, and B-4 on Metallic Materials for Thermostats, and for Electrical Resistance, Heating, and Contacts. The procedures have been forwarded to Committees E-1 and B-4 for their consideration. The efforts of the Cemented Carbide Producers Association to obtain standard cemented carbide hardness test blocks was reviewed by the committee. Their work, incomplete at this time, is considered essential in the reevaluation of the Recommended Practice for Hardness Testing of Cemented Carbides (B 294).

Section D on Friction Materials (H. B. Huntress, chairman) drafted the proposed Tentative Method of Test for Density of Sintered Metal Friction Materials, and the Tentative Method of Test for Determination of Transverse Rupture Strength of Sintered Metal Friction Materials, referred to earlier in this report. The committee is considering the interest shown in the thermal properties of sintered friction materials, and is of the opinion that ASTM already has adequate methods for the determination of specific heat. The committee is suggesting there is good correlation between electrical and thermal conductivity in sintered friction materials. Numerous cooperative test programs in the field of testing wet friction has been carried out. Significant disagreement in results has led to a study of exact procedures. A fixture for friction testing has been designed and it has been suggested that members of the committee use this specific design. The cost of this fixture is preventing its adoption. The dry friction testing program is continuing to use the Chase machine as a preliminary cooperative testing medium. The results of the first interlaboratory tests were revealing and it was decided to run cooperative dynamometer tests for comparison with the results from the Chase test. The committee feels that friction evaluation is much more qualitative than it is quantitative and therefore cannot predict if it will be possible to draft a test specification.

This report has been submitted to letter ballot of the committee, which consists of 79 members; 59 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. L. BONANNO,
Chairman.

C. G. JOHNSON,
Secretary.

REPORT OF THE JOINT ASTM-ASME COMMITTEE
ON
EFFECT OF TEMPERATURE ON THE PROPERTIES OF METALS*

Meetings:

Meetings of the ASTM-ASME Joint Committee on Effect of Temperature on the Properties of Metals, its panels and subcommittees were held during the past year in connection with the annual meetings of ASTM in Atlantic City, N. J., June 29, 1960, and The American Society of Mechanical Engineers in New York City, Nov. 30, 1960. The Panel on Structural Materials for Airframes and Missiles held a meeting in Los Angeles on Nov. 14, 1960.

Technical Sessions, Papers, and Publications:

A three-session symposium of 13 papers on Low-Temperature Properties of High-Strength Aircraft and Missile Materials was sponsored by the committee and presented on June 30, 1960, at Atlantic City, N. J., during the Annual Meeting of the Society. The symposium, organized by J. F. Watson of Convair-Astronautics, has been issued as a separate technical publication *ASTM STP No. 287*.

Two sessions of 12 papers dealing with high-temperature testing and data were also held during the Annual Meeting.[1]

An open panel discussion on "State of the Art in Design Applications of Low-Cycle Fatigue Data" was held by the Applied Research Panel at Atlantic City, N. J., on June 28, 1960. The panel consisted of L. F. Coffin, Jr., M. J. Manjoine, S. S. Manson, and W. Sarvent, who gave short presentations on various aspects of the subject.

A data compilation sponsored by the Joint Committee through the Data and Publications Panel, and compiled by J. W. Freeman and H. R. Voorhees of the University of Michigan, was published by ASTM early this year under the title "Elevated Temperature Properties of Aluminum and Magnesium Alloys" (*ASTM STP No. 291*).

Membership:

Mr. James Harlow, Philadelphia Electric Co., was elected to membership on the committee.

The resignation of Mr. A. W. Rankin, General Electric Co., was received during the year.

The committee records with sorrow the death of Charles T. Evans, Jr., who organized the Gas Turbine Panel and served as its chairman for several years. He was a member of the Technical Advisory Committee and served on the Executive Committee as a Member-at-Large.

Officers:

At the meeting of June 29, 1960, the following officers were elected for the term expiring June, 1962:

Chairman, J. J. Kanter, Crane Co.

First Vice-Chairman, R. A. Baker, Public Service Gas and Electric Co., N. J.

Second Vice-Chairman, G. V. Smith, Cornell University.

Secretary, H. C. Cross, Battelle Memorial Institute.

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* Presented at the Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] These papers have been published in *Proceedings*, Am. Soc. Testing Mats., Vol. 60 (1960).

TABLE I.—STATUS OF FUNDS IN THE ACCOUNT OF THE ASTM-ASME JOINT COMMITTEE ON EFFECT OF TEMPERATURE ON THE PROPERTIES OF METALS AS OF JANUARY 31, 1961.

Balance on hand, January 31, 1960	$86,112.22
Receipts, January 31, 1960 through January 31, 1961	20,568.04
Total	$106,580.26
Disbursements January 31, 1960 through January 31, 1961	41,252.63
Balance on hand, January 31, 1961	$65,427.63

Disbursement by Projects:

	Contract Term	*Contract Price*	*Paid*	*Balance*
Steam Power Panel:[a]				
SP-5, Use of Austenitic Alloy Steels in Steam Line Service (University of Michigan)	April 26, 1958 to Dec. 31, 1961	$47,500.00	$27,207.93	$20,292.07
SP-6, Use of Austenitic Stainless Steels in Superheater Tubes (University of Michigan)	Feb. 1, 1959 to Dec. 31, 1961	60,000.00	38,369.41	21,630.59
Data and Publication Panel:				
DP-10, Physical Properties of Metals (Battelle Memorial Institute)	Through Dec. 1, 1960	9,000.00	9,000.00	...
DP-11, -13, Punch Card Tabulation of Data (Battelle Memorial Institute)	Dec. 19, 1956 to Jan. 31, 1962	4,500.00	3,707.06	792.94
DP-14, Evaluation of Data for the Boiler and Pressure Vessel Code Committee	Sept. 1, 1960 to Aug. 31, 1961	3,500.00	731.02	2,768.98
Obligated				$45,484.58
Estimated uncommitted balance, Jan. 31, 1961				$19,943.05

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[a] It should be noted that contributions specifically in support of Projects SP-5 and SP-6 are anticipated but have not yet been received and so are not included in the above financial statement.

Members of Executive Committee: A. W. Rankin, General Electric Co., and R. C. Fitzgerald, Baltimore Electric Co.

Finances:

The status of funds in the committee's account as of December 31, 1960, is given in Table I.

ACTIVITIES OF PANELS AND PROJECT SUBCOMMITTEES

Finance Subcommittee (N. L. Mochel, chairman):

The campaign, initiated in June, 1959, with the objective of raising $150,000 had, as of November 30, 1960, resulted in total receipts of $125,440, with pledges of $41,500 yet to be received. The Finance Subcommittee reported excellent prospects of additional contributions of $45,000. The original goal is more than realized, and the promise of considerable additional support is a valid endorsement of confidence in the Joint Committee's importance and responsibilities to its sponsor societies.

Panel on Structural Materials for Airframes and Missiles (J. J. Heger, chairman:

Meetings of this panel were held in

June at Atlantic City, N. J., and in November at Los Angeles, Calif. The program and activities of the panel are organized as follows:

Task Group AM-3, Compression Properties of Sheet Materials.—This group, under H. C. Turner of Convair, has organized a symposium on Compression Properties of Sheet Materials to be presented at the 1961 Annual Meeting of ASTM at Atlantic City, N. J. The Test Methods Panel is cooperating with this group on the development of a recommended practice for determining compression properties of sheet materials at elevated temperatures and at conventional strain rates.

Task Group AM-4, Refractory Metals.—This task group, headed by Mr. S. E. Bramer, Culver City, Calif., conducted a survey on the problems pertaining to refractory metals. On the basis of this survey, the task group submitted to the Panel a recommended practice for reporting data on refractory metals. Revisions suggested by panel members were evaluated by the task group and a second draft will be circulated to the panel for approval by letter ballot. If approved, this recommended practice will be submitted to the Joint Committee for approval. If accepted, the task group plans to conduct a literature survey to determine the availability of data that meet the requirements of the recommended practice. The panel will then determine methods by which additional data, if shown to be needed by the literature survey, can be obtained.

The Task Group is encouraging work on the promulgation of ASTM standards for determining the inherent quality of refractory metal sheet products by ultrasonic techniques.

Task Group AM-5, Low-Temperature Properties of High-Strength Materials for Airframes and Missiles.—Under the chairmanship of J. F. Watson of Convair, San Diego, this task group organized a symposium of 12 papers on this subject as referred to earlier in the report.

Nonmetallic Materials.—Representatives of the panel are inquiring of other technical societies (for example, SAMPE) active in nonmetallic materials concerning problems in data dissemination and test methods in which the panel can be of assistance.

Problems Dealing with the Mach-3 Transport.—Morris Tiktinsky was appointed chairman of a task group to survey the problems dealing with structural materials for the Mach-3 transport. A report of this survey will be presented at the June, 1961, meeting of the panel. Harold Hessing of NASA discussed some of the materials problems of a Mach-3 Transport at the Joint Committee meeting on Nov. 30, 1960.

Data and Publications Panel (George V. Smith, chairman):

This panel met on June 27 and again on Nov. 29, 1960, and the status of its activities is summarized as follows:

Project DP-9, Survey of Aluminum and Magnesium Alloys.—This survey was completed and published under the title, "Elevated-Temperature Properties of Aluminum and Magnesium Alloys," *ASTM STP 291.* This volume contains 314 pages of tabular and graphical data.

Project DP-10, Effect of Temperature on Selected Physical Properties of Metals.—This project, under contract with Battelle Memorial Institute, was completed and the results published as *ASTM STP No. 296* under the title "Physical Properties of Metals and Alloys from Cryogenic to Elevated Temperatures." The data, presented in tabular and graphical form, cover the following metals and their alloys: aluminum, cobalt, iron, magnesium, molybdenum, and nickel; references are made to other pertinent compilations.

Project DP-13, Continuing Data Sur-

vey and Punched Card Tabulation of Data.—The first phase of this program, which is designed to furnish, on punched cards, high-temperature strength data for metals and alloys was completed with the announcement by ASTM that punched cards were available for purchase. The cards are packaged and sold in sets of the following categories:

Group I.—Light Alloys.

Group II.—Iron and Steel, and AISI Stainless.

Group III.—Superalloys, Refractory Alloys.

The initial distribution consists of 25 cards in each of Groups II and III, plus a Miscellaneous Group that cuts across all three categories. Subsequent sales will be confined to the three mentioned groups only. The panel is continuing its solicitation of data from testing laboratories and will subsequently issue cards confined to the three groups only.

Project DP-14, Evaluation of Data for the Boiler and Pressure Vessel Code Committee.—This project was established as a result of a request for data evaluation from the Subgroup on Metals Engineering of the ASME Boiler and Pressure Vessel Committee's subcommittee on Ferrous Materials and High-Temperature Alloys. This subgroup is charged with the responsibility of evaluating materials as to their suitability for use, and with establishing working stresses. The Data and Publications Panel was requested to screen existing data and data that will be compiled under Project DP-13, to establish whether they fall within the variance limits of appropriate specifications and, if so, to present the data in desirable form for ready evaluation by the Subgroup on Metals Engineering. The Joint Committee appropriated $3500 to defray the costs of this work during the first 6 months at Battelle Memorial Institute, where the work on Project DP-13 is centered.

Available data for stainless steel types 316 and 321 have been evaluated and submitted to Battelle. Data on types 304L and 316L, particularly short-time data for temperatures up to 1000 F, are also expected to become available for the analysis.

Applied Research Panel (W. Lee Williams, chairman):

A meeting of this panel was held on June 28, 1960, under the chairmanship of A. W. Rankin, of General Electric Co., who recently resigned and has been replaced in this capacity by W. L. Williams, U. S. Naval Engineering Experimental station.

Inquiries were sent to several laboratories asking for proposals on investigations on "The Effect of Surface Imperfections on Thermal Fatigue." Only the general goal and outline of this project had been outlined in the inquiries, leaving it to the individual laboratories to propose programs around these goals with an estimate of the costs involved.

A considerable amount of information on rupture tests of longer than 25,000 hr was obtained. Most of these data are of European origin and not on American compositions. The European laboratories conducting these tests will be approached again with regard to short-time tests on the same alloys which might be used to evaluate parameter extrapolations. If suitable short-time data are not planned by those laboratories, they will be requested; however, if these laboratories are not interested in making such tests, they will then be requested to furnish material to the panel so that it can make the required tests. In the latter case, some money will be needed, the amount of which is not known.

Rupture tests of longer than 50,000 hr are being planned on the type 304 material available at the Bureau of Standards for rupture-test standardization.

An open panel discussion on the subject "State of the Art in Design Applications of Low-Cycle Fatigue Data" was held during the 1960 Annual Meeting of ASTM.

The present chairman of this panel, W. L. Williams, is reviewing the objectives and scope of this panel and contemplates recommendations for its reorganization to the Technical Advisory Committee.

Gas Turbine Panel (*Bruce O. Buckland, chairman*):

Meetings of the Gas Turbine Panel were held on June 27 and Nov. 28, 1960. A task force reported on efforts to stimulate more aircraft gas turbine participation, with particular emphasis on commercial airline operators. Four of the five major airlines have indicated interest and a follow-up will be made.

A second task force assigned to seek greater participation on the part of the industrial gas turbine users will report at the next meeting of the panel.

A third task force reported the results of a study of the present state of knowledge on creep damage appraisal of gas turbine components, especially turbine blades. This information would be extremely useful in attempting to determine how much life is left in a turbine bucket, for example, after a period of service, and whether parts can be rejuvenated by suitable heat treatment. On the basis of the task force report and the vigorous discussion that followed, a new task force was named with suitable representation from the various types of interest in the panel. This task force will try to obtain information pertinent to the subject and, if appropriate, to recommend to the panel a symposium or other medium to get the information into usable form and to recommend any research indicated by this work.

Another problem receiving the attention of the panel is that involving thermal fatigue of critical parts. A task force was set up to review the information now available, but more particularly to appraise what use has been made of the considerable quantity of information already published in this area.

Two other problems were brought to the attention of the panel, one concerned with erosion of turbine blades in service and the other with repair welding of gas turbine components. Appraisal committees were selected for each of these areas and instructed to determine whether a problem exists and whether task forces should be set up.

Low-Temperature Panel (*A. L. Tarr, chairman*):

Meetings of the panel were held on June 27 and Nov. 28, 1960. A symposium including 12 papers has been arranged for the June ASTM meeting under the title "Evaluation of Metallic Materials in Design for Low-Temperature Services."[2]

Liaison and coordination is maintained with other groups concerned with low-temperature effects and data collection. The National Bureau of Standards' Monograph 13, "Mechanical Properties of Structural Materials at Low Temperatures," is now available for purchase from the Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C. Also, the Cryogenic Engineering Laboratory of the National Bureau of Standards is now preparing for publication a "Compendium on Properties of Materials at Low Temperatures" in three parts: Part I for Fluids, Part II for Solids, and Part III to contain the bibliography of references and other miscellaneous information. The Cryogenic Data Center, established in 1958, at the NBS Cryogenic Engineering Laboratory is endeavoring to index as much of the world's cryogenic literature as possible and store the references for easy retrieval by machine storage and sorting. Attention is called to the 1960 Cryogenic Engineering Conference which is being cosponsored by the University of Colorado and the National Bureau of Standards and which will be held in Boulder, Colo. on Aug. 23, 24, and 25. Attention is also directed to the "Air Weapons Materials Application Handbook Metals and Alloys," *ARDC TR 59-66*, a voluminous compilation edited by G. Sachs, Syracuse University Research Inst. under sponsorship of the Air Research and Development Command, United States Air Force. The purpose of this handbook is to consolidate into one volume the latest and most up-to-date data on the properties of metals and alloys of primary importance for the production and design of air weapons. Part I of the handbook contains a general discussion of the properties which are presented in detail for each alloy in Part II. Requests for this document should be addressed as follows:

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[2] To be published as *ASTM STP No. 302.*

Commander
Air Research and Development Command
Washington 25, D. C.

Attention: Mr. C. W. Kniffin, RDR-2

Petroleum and Chemical Panel (*C. L. Clark, chairman*):

This panel held a meeting on Nov. 28, 1960. Its active projects had been concluded as reported previously, but discussion was opened on future work. The most pressing problem appeared to be the lack of stress-rupture data in the temperature range of 1800 to 2000 F on materials suitable for the manufacture of ethylene. It was decided to formulate a program designed to provide the necessary information on several selected alloys in the 1800 to 2000 F range and that the work should be conducted on a contract basis.

Two separate projects are being organized as follows:

1. A testing program to include the following:

(*a*) Stress-rupture tests at 1800, 1900, and 2000 F for times extending to 10,000 hr;

(*b*) Selection of alloys to include: cast alloys, types 309, 310, and modifications of 310 containing cobalt, tungsten, molybdenum, etc.; wrought, types 309 and 310, and Incoloy, welded, Hastelloy X; and

(*c*) Bids to be requested from institutions or organizations interested in doing the work.

2. A proposal outlining a program aimed at determining surface stability characteristics of these alloys in the range of 1800 to 2000 F.

Steam Power Panel (*Paul Brister, chairman*):

The panel held meetings on June 28 and Nov. 29, 1960.

Project SP-5, Use of Austenitic Steels in Main Steam Line Service.—A prodigious effort has been made by the project committee, which has held a number of meetings and conferences during the year. An extended status report on Project SP-5 contracted to the University of Michigan is appended to this report.[3] The committee is continuing to support a high level of activity on Project SP-5 through the year 1961.

Project SP-6, Use of Austenitic Stainless Steel in Superheater Tubes.—The work on this project has been directed at identifying the factors which affect the high-temperature strength properties of type 321-H steel. This project, under contract at the University of Michigan is in its third year and is to be broadened to cover more completely types 304, 316, and 347. The results of this project are of considerable importance to the support of allowable stresses under the ASME Boiler Code and the ASA Code for Pressure Piping. An extended status report on Project SP-6 is appended to this report.[4]

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[3] See p. 378.

[4] See p. 382.

Project SP-7, Material Evaluation.—The purpose of this project is to evaluate new materials for use at high temperatures. A survey of present power plants operating at 1100 F or higher is being made to determine what materials are available that would have higher strength than those now in commercial use. Initial discussions considered primarily the problems in the boiler, and pointed out that tube wastage (external loss of metal, corrosion) is a very serious problem at high metal temperatures in combustion gas streams. Materials presently considered for study are: types 316 Cb, G 19, 17-14 CrMo, 15-15 N, and N-155. Information on oxidation and corrosion properties as well as creep and stress rupture data is to be developed.

Project SP-8, Effect of Long-Time Service on Elevated Temperature Properties of Tubes and Pipes.—A program has been developed to evaluate long-time effects as follows:

1. Obtain for various utilities samples for study of materials with at least 10 yr service,
2. Implement a test program for evaluation of the materials removed from service, and
3. Settle upon means for evaluating the information on loss of impact strength, as reported in a paper by E. A. Sticha titled "Structural Stability of Commercial Wrought Austenitic Steels for Power Plant Piping to 1450 F."[5] This will be considered in conjunction with other properties to be studied.

There is considerable concern over the significance of the information in the Sticha paper, particularly with regard to the fact that there are many factors not covered in the paper that need consideration. Project SP-8 will not be in a position to make pertinent comment for some time; the views of individuals with information on this subject are sought.

Because of the controversial aspect of the subject, consideration is being given to a session or symposium on the subject of effect of time and temperature on the properties of tubes and pipes under the title "Effect of Long-Time Service on Elevated Temperature Properties of Tubes and Pipes."

Test Methods Panel (J. W. Freeman, chairman):

The panel held meetings June 27 and Nov. 29, 1960. Its program may be summarized as follows:

An extensive survey has been conducted among members of Aerospace Industries Assn. of their experience with the Tentative Recommended Practices E 150[6] and E 151.[7] The practices were found to be generally satisfactory. Most criticism arose from attempts to adapt the methods to simulated service testing. It does appear that the tolerance of specimen dimensions and possibly the temperature limits should be considered for revision before the practices are adopted as standard.

There was urgent need for recommended practices covering compression testing with high heating rates and compression, bearing, and shear creep testing. Drafts will be prepared based on the information that will be developed on these subjects at symposia at the 1961 Annual Meeting of ASTM.

A practice for the determination of dynamic and static moduli values is being prepared jointly with Subcommittee III on Elastic Properties and Definitions on Mechanical Testing of ASTM Committee E-1 on Methods of Testing.

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[5] *Power Magazine* (1960).

[6] **Tentative Recommended Practice for Conducting Creep and Creep-Rupture Tension Tests of Metallic Materials Under Conditions of Rapid Heating and Short Times (E 150 – 59 T), 1959 Supplement to Book of ASTM Standards, Part 3, p. 50.**

[7] **Tentative Recommended Practice for Tension Tests of Metallic Materials at Elevated Temperatures with Rapid Heating and Conventional or Rapid Strain Rates (E 151 – 59 T). 1959 Supplement to Book of ASTM Standards. Part 3, p. 59.**

Under a project initiated by the late C. T. Evans, Jr., specimens for the creep-rupture test specimen bank are in the hands of cooperating laboratories for a trial test of the material. A paper by W. D. Jenkins, W. A. Willard, and W. J. Youden of the National Bureau of Standards on the original evaluation of the material was published in *Materials Research & Standards*, Vol. 1, No. 2, pp. 104–108, Feb., 1960, under the title "A Report on Stress Rupture Tests at 1350 F on Type 304 Stainless Steel."

A task force on notched-specimen rupture testing does not consider it advisable to set up an acceptance type testing practice or one which is presumed to differentiate between a notch-ductile and notch-brittle material. Instead, it is proposed to set up a type notch-acuity specimen (or possibly several types), to discuss preparation of notched specimens, and comment on utilization of notched specimen rupture data. Difficulties have been experienced in agreeing on notch measurements. There is a feeling that it will be desirable to standardize on a test specimen (or on several specimens), modifying the recommended practice as more information is developed.

Laboratories using Recommended Practices E 21[8] and E 139[9] are being surveyed as to the acceptability of the methods before proposing that these practices be adopted as standard.

At the request of Aerospace Industries Assn., the Panel is studying the need for a practice for notch testing to establish transition temperature.

International Conference on Creep and Fracture (N. L. Mochel, secretary):

Under the joint sponsorship of the ASME Metals Engineering Division and ASTM, an International Conference is scheduled to be held at the Hotel Biltmore, New York, N. Y., August, 1963.

N. L. Mochel, W. E. Leyda, and R. F. Miller, representatives for the Joint Committee, attended a High-Temperature Metals Conference at Düsseldorf in June, 1960, and took advantage of this occasion as well as subsequent contacts in England to obtain agreements and understanding in behalf of the 1963 Symposium.

Respectfully submitted on behalf of the committee,

J. J. KANTER,
Chairman.

HOWARD C. CROSS,
Secretary.

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[8] Tentative Recommended Practice for Short-Time Elevated Temperature Tension Tests of Materials (E 21 - 58 T), 1958 Book of ASTM Standards, Part 3, p. 217.

[9] Tentative Recommended Practice for Conducting Creep and Time-for-Rupture Tension Tests of Materials (E 139 - 58 T), 1958 Book of ASTM Standards, Part 3, p. 224.

EDITORIAL NOTE

Subsequent to the Annual Meeting, the Joint Committee on Effect of Temperature on the Properties of Metals presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Recommended Practice for:

Conducting Creep-Rupture Tension Tests of Metallic Materials Under Conditions of Rapid Heating and Short Times (E 150 - 59 T), and

Tension Tests of Metallic Materials at Elevated Temperatures with Rapid Heating and Conventional or Rapid Strain Rates (E 151 - 59 T).

These recommendations were accepted by the Standards Committee on September 12, 1961, and the revised tentative recommended practices appear in the 1961 Book of ASTM Standards, Part 3.

APPENDIX I

SUMMARIZED STATUS REPORT ON BASE-METAL CRACKING ADJACENT TO WELDS IN TYPE 347 STEAM LINES

Report of Project SP-5 of Steam Power Panel, ASTM-ASME Joint Committee on the Effect of Temperature on Properties of Metals

This report summarizes the research conducted, the results obtained, and the proposed future of the investigation as of October, 1960. The general objective has been to determine the causes for cracking in the base metal adjacent to the weld as a result of welding steam lines made from type 347 (18-8 + Cb) stainless steel. The reports of service experience indicated a range in performance from freedom from cracking to extreme sensitivity to cracking. The cracking was often evident during the welding operation, although in many cases it did not become evident until after varying periods of service.

When the research program was started, the Steering Committee for Project SP-5 directed that the investigation concentrate on the problem of cracking as a result of welding. After consideration of initial results, it was further directed that the causes for high and low hot ductility in hot-ductility testing be determined. This was based on the assumption that there were distinct metallurgical differences between such materials. Furthermore, low ductility after heating to relatively low maximum temperatures in this test using conditions simulating heating during welding was believed to be indicative of susceptibility to cracking.

Research Program

The research program was developed along the following lines:

1. Development of a laboratory procedure for estimating susceptibility to cracking as a result of welding.

2. Research on the reasons for low and high ductility between heats in hot-ductility testing. The following steps were involved:

(*a*) A limited study of the influence of conditions of hot working and heat treatment prior to testing.

(*b*) A study of the influence of trace elements.

(*c*) Based on the observation that low ductility was associated with the appearance of a "eutectic-like" microstructure not dependent on trace elements, investigations were started to establish the phases associated with temperature of appearance of the "eutectic-like" structure.

(*d*) The results of (*c*) led to investigation of the influence of carbon and columbium content on the phases formed and the temperature of appearance of the "eutectic-like" structure.

Test Results

An initial group of specimens taken from steam line service was classified in accordance with reported service experience by rapidly heating to a maximum temperature of 2450 F, cooling to 2300 F, and quickly breaking the specimens. Those specimens which had a history of cracking had low ductility and those free from cracking had high ductility. This procedure was based on the test developed by Professor Nippes at Rensselaer Polytechnic Inst. in which a series of specimens are heated under simulated welding conditions and pulled apart so as to establish curves of ductility versus temperature. In general, those materials with low hot ductility showed low reduction of area when heated to 2350 to 2450 F, cooled to 2300 F, and broken.

The study of the causes for the differences between specimens of type 347 stainless steel which influence the results of hot-ductility tests gave the following results:

1. Low ductility was always associated with the appearance of a "eutectic-like" structure in the microstructure. This has been verified for specimens from RPI type hot-ductility testing machines as well as the modified test used at the University.

2. Based on this observation, the research at the University subsequently used the lowest temperature of appearance of this structure as a sorting test for hot-ductility characteristics. Specimens $\frac{1}{4}$ by $\frac{1}{4}$ by $\frac{3}{8}$ in. are placed in a furnace at the proper maximum temperature and held 1 min, air cooled, and examined metallographically. This procedure has the main advantage of simplified accurate pyrometry and low cost. The holding period of 1 min evolved from the observation that prolonging the exposure time intensified the differences between lots of the steel. The furnace used gave heating rates similar to those in hot-ductility testing.

3. Those specimens of type 347 steel with low hot ductility were found to develop the "eutectic-like" structure after relatively low maximum temperatures of heating. Those with high ductility required higher temperatures of heating. Incomplete data suggest that poor service experience is associated with the appearance of the structure at temperatures below about 2440 to 2450 F. The temperature has been found to be lower with increasing severity of cracking. In at least one borderline case, the temperature of appearance of the structure (and low ductility) was somewhat time-dependent. High rates of heating gave high ductility. Somewhat lower rates resulted in low ductility. The two lots of wrought pipe studied intensely because they initially indicated low and high ductility differed mainly in that the temperature of appearance of the structure could be reduced to lower temperatures for the low ductility material by slightly reducing the rate of heating. These findings suggest the following:

(*a*) Cracking can be minimized by keeping the heat input during welding at a minimum. This is reflected in experience that small electrodes with minimum power reduce cracking.

(*b*) In borderline cases, cracking experience can be highly dependent on the conditions of welding.

4. The study of the influence of hot working and heat treatment indicated that unidentified heat-to-heat differences were more influential than these variables. The relatively small differences between the two lots of materials combined with the consequent sensitivity to test conditions may, however, have obscured effects of these variables.

5. The study of trace elements indicated that it was not necessary to add such elements to develop differences in the temperature of appearance of the "eutectic-like" structure.

6. Current incomplete research indicates that:

(*a*) The temperature of appearance of the "eutectic-like" structure is reduced by high columbium in relation to the carbon content within the type 347 range of composition. Low carbon, in itself, may also be responsible.

(*b*) When the temperature of appearance of the structure is less than 2440 to 2450 F, Fe_2Cb has been identified in the structure. To do so, it has been necessary to heat to higher temperatures than the lowest temperature for its appearance to increase the amount of "eutectic-like" structure. Except in one cast specimen with very poor cracking experience and a laboratory heat with high columbium, Fe_2Cb has not been identified without heating to develop the "eutectic-like" structure. The results suggest a ternary eutectic of austenite, CbC, and Fe_2Cb, although there are unexplained observations which do not fit this theory.

(*c*) Incomplete data suggest that service experience may correlate with the carbon and columbium content.

(*d*) If the carbon:columbium relation proves correct, the necessary restrictions of these elements would be within practical limits.

7. The correlations of temperature of appearance of the "eutectic-like" structure and carbon and columbium content with reported hot-ductility data have not been good. This may be due to a reduction in liquation temperature under the extremely fast heating rates used, or to problems of temperature measurement. The temperatures required to develop "eutectic-like" structure under the conditions of the test used at the University have been verified independently by two other laboratories which heat treat in the range of 2400 to 2500 F as a matter of routine.

SUMMARIZED CONCLUSIONS

1. Low hot ductility is associated with the appearance of a "eutectic-like" structure.

2. Low temperatures for the appearance of this structure are associated with low hot ductility and cracking during welding. The question of its relation to cracking originating during service has not been investigated.

3. Procedures which minimize the maximum temperature and time of heating of the base metal during welding reduce cracking.

4. Incomplete tentative data suggest that low hot ductility is associated with high columbium content in relation to the carbon content. Apparently, this allows the formation of Fe_2Cb during heating for welding which, in combination with the austenite and CbC, develops a ternary eutectic which can have a relatively low temperature of formation. Highly tentative results, which if verified, suggest that the restriction of columbium in relation to carbon would be within practical limits.

5. To date, the data indicate that trace elements, prior processing, and heat treatments have a minor influence in comparison to the carbon-columbium effect.

6. Until these factors are cleared up, it will not be possible to specify type 347 for main steam lines with assurance of freedom from cracking.

PROPOSED FUTURE RESEARCH

The main emphasis of proposed future research is to concentrate on a study designed to determine if there are conditions which can lead to service difficulties as the result of welding of steam lines made from types 316 and 304 stainless steels. Compositional, manufacturing, and heat treatment variables will be considered from the viewpoint of cracking during welding and any features of the influence of welding on possible difficulties during service in types 304 or 316. This proposed research recognizes that the problem of cracking during welding appears to be slight on the basis of experience to date. The objective will be to determine if there are conditions where this is not true and, particularly, to determine if the welding is liable to cause conditions which during subsequent service may lead to failures which could be avoided by adequate forewarning.

The research on type 347 should be continued to establish definitely the role of carbon and columbium content and to survey the influence of variations of chromium, nickel, manganese, and silicon within the specification range. This will be done by defining their influence on the temperature of appearance of the "eutectic-like" structure and establishing the role of Fe_2Cb more definitely. A limited amount of research should also be carried out to clarify the influence of conditions of the metal in the heat-affected zone on subsequent cracking during service, either as a result of cracking initiated during service, or as a result of propagation of minute cracks introduced during welding.

It is expected that the research can be completed during 1961.

APPENDIX II

SUMMARIZED STATUS REPORT ON STAINLESS STEEL SUPERHEATER TUBING

Report of Project SP-6 of Steam Power Panel, ASTM-ASME Joint Committee on the Effect of Temperature on Properties of Metals

The over-all objective of Project SP-6 is to delineate the variables of composition, processing, and heat treatment influencing the properties of stainless steels as superheater tubing so that optimum performance in service can be obtained. It is expected that the results will be useful in the development of materials capable of improved performance for the future.

The research to date has been carried out using type 321 (18-8 + Ti) as the main experimental alloy. Limited work has been initiated using type 304 (18-8) steel. Type 321 was selected for the initial research because it is currently widely used as superheater tubing and because it is an ideal material for experimental purposes due to marked response of its properties to the variables to be investigated.

Experimental Program

Upon the advice of the Steering Committee for Project SP-6, the following experimental program has been carried out:

1. The response of type 321 superheater tubing to final heat treatment has been defined. Production tubing from two sources was provided by the manufacturers as produced ready for final heat treatment after the final cold-working operation. Both tube-reduced and cold-drawn tubing were used. Additional samples have been supplied as fully heat treated by manufacturers, and experimental heats have been made in the laboratory to introduce special variables.

2. The creep and rupture properties at 1200 F have been used to evaluate the influence of temperature of heat treatment ranging from 1600 to 2300 F. Appropriate structural studies have been carried out to delineate the cause of the observed effects.

3. The influence of carbon content has been defined. The results from tubing have been supplemented by laboratory vacuum induction melted heats.

4. To assist in establishing the reasons for the variation in properties of type 321 superheater tubing with heat treatment, an investigation of the influence of reheating at lower temperatures after final heat treatment has been carried out. A limited number of rupture tests at 1300 and 1400 F have been conducted for the same reason.

5. Tension tests at 1200 F have been used to evaluate the state of the carbon as an aid in defining the strengthening mechanism.

6. Because type 321 steel develops extensive sigma phase during exposure to high temperatures, particularly when initially fine grained, a study was made of the significance of this reaction.

7. A large number of samples of tubing from service were examined and

tested to study the influence of exposure times beyond those which could be obtained in the laboratory within the time available.

8. A study of the response of type 304 tubing to heat treatment is in progress to obtain data which will be used to supplement and contrast with that for type 321 to assist in clarifying the metallurgical principles for austenitic stainless steel superheater tubing.

SUMMARY OF RESULTS

The results being reported are, in most cases, based on stress-rupture time curves at 1200 F, with the longest tests generally being of the order of 1000 to 2000 hr. In some cases the data are incomplete and reflect trends.

Influence of Temperature of Heat Treatment:

1. The rupture strength increases and ductility decreases with temperature of heat treatment up to a temperature governed by the carbon content. For a carbon content of 0.04 per cent this temperature is about 2000 F. To obtain higher strength from higher temperatures of heat treatment the carbon content must be higher.

2. Creep tests of 5000 hr duration at 1200 F under the present ASME Boiler Code stress of 5000 psi indicate that the temperature of heat treatment must be at least 2000 F to develop the expected creep strength.

Heat-to-Heat Variations:

There are unidentified heat-to-heat variations which influence the level of strength which are apart and distinct from the general rules cited in (1) above. Thus, two heats with 0.05 per cent carbon will develop their own maximum strength when heat treated at about 2050 F.

Tube-Reduced Versus Cold-Drawn Tubing:

No major difference was found between tube-reduced and cold-drawn tubing for the one heat of material studied.

Influence of Carbon Content:

The data indicate that the major factor governing creep-rupture strength is the amount of carbon and titanium in solution at the start of the test. The amount which can be in solution increases with temperature of heat treatment. When the temperature of heat treatment is 2000 F or lower, the creep-rupture strengths of heats with carbon contents ranging from 0.04 to 0.10 per cent will be largely independent of the carbon content. If the temperature of heat treatment is increased above 2000 F, the strength of the material with 0.04 per cent carbon will not be further increased but those with higher carbon will be. The degree of increase will be limited by the carbon content so that when the temperature of treatment approaches 2300 F, only the higher carbon heats continue to improve. Rather high strengths result from combinations of high carbon and high temperatures of heat treatment.

Influence of Titanium:Carbon Ratio:

The influence of the relative levels of titanium and carbon has not been adequately investigated. Preliminary data suggest that the titanium:carbon ratio should be about 4:1 (sufficient to form TiC). If the ratio is lower or higher, the increase in strength will probably be in accordance with the amount of carbon and titanium forming TiC.

Influence of Grain Size:

Grain size in itself has little effect on strength. Ductility is lower, however, for a given strength, the coarser the grain size. The major effect is the degree of solution of titanium and carbon. Thus, grain size will reflect strength only to the degree that it correctly reflects solution of titanium and carbon. Incomplete data suggest the following trends:

1. A coarse grain size with a low carbon content will have low strength even though the temperature of heat treatment is high.

2. Material with a coarse grain size can be heated at lower temperatures and its strength reduced as a result of the subsequent precipitation of TiC.

3. When tubing is given the final cold-working operation and then heat treated, a coarse grain size is indicative of adequate solution of titanium and carbon when the carbon content is greater than 0.04 per cent.

4. There appears to be an indeterminate region just before the temperature of heat treatment is high enough to cause grain coarsening. The degree of solution of titanium and carbon may be adequate to develop acceptable strengths. The combination of some solution plus the high ductility from the fine grain size can result in acceptable rupture strengths with somewhat low creep strengths. There is the additional probability that other factors than solution of TiC govern the grain-coarsening characteristics. Possibly the strength will tend to come up without grain coarsening when the titanium and carbon contents are high enough to allow sufficient undissolved TiC to restrict grain growth at the temperature of heat treatment.

Metallurgical Principles Governing the Properties of Type 321:

The following results have been obtained toward clarification of the metallurgical principles governing the properties of type 321 stainless steel:

1. Titanium and carbon need to be in solution at the start of a test to be effective in increasing strength.

2. If either titanium or carbon is absent from the composition (or sufficiently low), the rupture strength becomes nearly independent of heat treatment.

3. TiC precipitates rapidly when recrystallization occurs at temperatures below the solution temperature. A temperature of about 2000 F is required to place about 0.04 per cent carbon in solution. Thus, where cold-worked material is heat treated at 1600 to 1800 F, there is extensive precipitation of TiC in normal type 321 steel. The TiC is in the form of fairly massive particles even though the time of heat treatment is normally only ½ hr. Due to the extensive precipitation of TiC in this form, the strength is low and becomes lower the lower the temperature of heat treatment. The strength is low due to the precipitation of the TiC and not due to the fine grain size.

4. Fully solution-treated material is reduced in strength by reason of the precipitation of TiC which accompanies reheating to temperatures below the solution temperature. The time required for a given reduction in strength increases as the temperature of reheating is lowered. Only a short time is required at 1750 F. This increases to about 50 hr at 1600 F and becomes hundreds of hours at lower temperatures. The reduction in strength, however, does not bring the strength down to as low a level as when recrystallization of the cold-worked material occurs during heat treatment at 1600 to 1800 F.

5. In no case has an increase in creep-rupture strength been found from reheating below the solution temperature before testing. The yield strength and hardness can be raised substantially by prior heating below 1600 F, but the rupture strength decreases.

6. The decrease in rupture strength by the prior precipitation of TiC is accompanied by marked increase in ductility in the rupture tests.

7. Rupture tests conducted at 1300 and 1400 F show that strength is highly dependent on prior temperature of solution treatment. Thus, the reactions which take place are effective in influencing strength at these temperatures even though prior exposure at these temperatures reduces strength when subsequently tested at 1200 F.

8. When solution treated, the stress-strain curves at 1200 F showed marked serrations. Prior heating reduces these serrations and, when the strength is lowest, they entirely disappear. This indicates that carbon and/or nitrogen in solution are responsible for both serrated stress-strain curves and elevated creep strength. In the absence of titanium there is a marked decrease in both the serrated nature of the stress-strain curve and creep strength even when solution is complete. This is evidence that some interaction between titanium and carbon prior to the formation of TiC is responsible for elevated creep strength which is partially reflected in the serrated nature of the stress-strain curve.

Role of Sigma Phase:

The results of the study of sigma phase formation indicate that it is an aftereffect. No evidence was found to indicate that it was a major contributor to low strength. Apparently large grain size reduces the rate of formation of sigma to a marked extent.

Type 304 (18-8) Stainless Steel:

The data on type 304 stainless steel are still not sufficiently complete to draw extensive conclusions. The following results have been obtained:

1. The temperature of heat treatment has comparatively little effect on rupture strength. Increasing the temperature of heat treatment reduces ductility, and when very high can consequently reduce rupture strength.

2. Type 304 has lower short-time strength than type 321. Extrapolation of the stress-rupture time curves indicates long-time strengths equal to the type 321 strengths on the high side of the band. Limited data suggest that the insensitivity to heat-treatment temperature is due to the type of carbide which forms. In type 304 complex carbides form, while the preferential interaction between titanium and carbon in type 321 tends to strengthen that material. No information has yet been obtained to explain the maintenance of strength of type 304 at long-time periods, while type 321 tends to exhibit a more rapid drop in strength.

CONCLUSIONS

The following conclusions are based on the research on type 321 stainless steel using rupture tests at 1200 F for a basis of evaluation unless otherwise indicated:

1. The research substantiates the minimum 0.04 per cent carbon and minimum temperature for heat treatment of 2000 F presently specified for type 321-H.

2. Prolonged creep tests under the present ASME Boiler Code stress of 5000 psi at 1200 F indicate that a minimum temperature of 2000 F for heat treatment is required to develop expected creep strength.

3. The rupture strength increases with temperature of heat treatment independent of carbon content up to the point where all the carbon is in solution. About 0.04 per cent carbon can be dissolved at about 2000 F. The strengths will be independent of the carbon content up to this point as long as it is at least 0.04 per cent. Further strengthening can be obtained with higher carbon contents by raising the temperature of heat treatment. Extremely high strengths result from combinations of high carbon and high temperatures of heat treatment.

4. While no deviations have been found for the preceding generalities, it has been found that there are unidentified heat-to-heat differences which result in

differences in level of strength for a given heat treatment and carbon content.

5. Strength depends on carbon and titanium being in solution at the start of exposure to stress.

6. Reheating at lower temperatures after solution treatment causes precipitation of TiC and, therefore, lowers strength.

7. The way in which titanium and carbon combine to increase strength when initially in solution is not yet clear. If either is absent, the strength is low regardless of the temperature of heat treatment. Prior heating to form TiC lowers strength. Yet, if both are in solution at the start of a test, they markedly increase strength. This holds at test temperatures as high as 1400 F.

8. Grain size in itself has only a minor effect on rupture strength. Whether or not grain size reflects strength depends on whether it correctly reflects the degree of solution of titanium and carbon. Ductility in the rupture test is lower the coarser the grain size for a given level of strength.

9. The appearance of sigma phase appears to be a reaction which has little effect on the strength level. Increasing the grain size appears to retard sigma formation.

10. As far as can be ascertained, the manufacturing procedure, such as tube reducing versus cold drawing, has little influence on properties of type 321.

11. Results obtained to date on type 304 (18-8) superheater tubing indicate that:

(*a*) The rupture properties at 1200 F are nearly independent of the temperature of heat treatment. The only exception yet found is very high-temperature treatments which reduce ductility and thereby reduce rupture life.

(*b*) Type 304 has lower short-time strengths than type 321 which has been adequately solution treated. The long-time strengths of type 304 can, however, be equal to properly heat treated type 321.

(*c*) The data suggest that the cause for the difference in response to heat treatment between types 304 and 321 is the type of carbide formed.

FUTURE RESEARCH

1. Use the remaining time under the present contract to analyze data and prepare a technical report on the results to date. Experimental work will be limited to laboratory structural studies.

2. Future research on type 321 will be limited to completion of the work necessary to fill in gaps in the data and to such final steps as the work completed to date may indicate. In particular, it is considered essential to complete the establishment of the metallurgical principles governing the strengthening of type 321, including the identification of the cause of the heat-to-heat variation.

3. The research on type 304 should be extended to include more temperatures of heat treatment, creep strength as well as rupture strength, and to verify the results on material made by another manufacturer. The research should include the structural studies and the special experiments necessary to clarify the metallurgical principles governing the properties of type 304 steel.

4. During the course of the next year the metallurgical principles should be well enough established so that predictions should be possible for other alloys. This will be done with a view of checking them on types 316 and 347 or on such experimental alloys as may, in the judgment of University representatives and the Steering Committee, be justified. In particular, the principles are to be evaluated in terms of the possibility of producing alloys with better properties than those of current standard stainless steels.

5. It is expected that this work can be completed during 1961.

REPORT OF ADVISORY COMMITTEE
ON
CORROSION

The Advisory Committee on Corrosion held two meetings during the year: on June 28, 1960, in Atlantic City, N. J., and on January 31, 1961, in Cincinnati, Ohio.

PERSONNEL

New committee representatives are as follows: for Committee A-3, R. A. Clark and C. F. Walton replace T. E. Egan and H. W. Stewart; for Committee C-19, Steven Yurenka and Dan Miller replace T. P. Pajak and H. F. Zemer; for Committee D-14, A. A. Marra replaces J. E. Rutzler, Jr.; and for Committee D-20, J. B. DeCoste and A. Mendizza replace F. W. Reinhart and J. H. Gibbud.

Committee E-1 on Methods of Testing has requested representation on the Advisory Committee and has designated A. C. Webber and F. M. Gavan as representatives.

Mr. C. P. Larrabee has been recommended for reappointment as a member-at-large for a three-year term beginning June 1961.

The officers elected for the ensuing term of two years are as follows:

Chairman, C. P. Larrabee.
First Vice-Chairman, C. H. Sample.
Second Vice-Chairman, W. H. Ailor.

STATUS OF ATMOSPHERIC EXPOSURE PROGRAMS

Committee A-5 on Corrosion of Iron and Steel:

Aluminum- and zinc-coated hot-dipped steel roofing sheets (28 by 36 in.) were exposed in September, 1960 at State College, Pa.; Newark (New York area), N. J.; Kure Beach (800-ft site), N. C.; Point Reyes, Calif.; and Freeport, Tex.

Aluminum- and zinc-coated wires and fabricated fencing are being exposed at State College, Pa.; Kure Beach (80- and 800-ft), N. C., Newark (New York area), N. J.; Point Reyes, Calif.; Freeport, Tex.; and Manhattan, Kans.

Inspection reports of four atmospheric programs are appended to the 1961 Annual Report of Committee A-5: aluminum- and zinc-coated hardware exposed in 1958; various coatings on hardware exposed in 1928; zinc-, lead-, and copper-coated steel wire and stainless steel wire exposed in 1936; and galvanized roofing sheets exposed in 1926.

A comprehensive study of the twenty-year data from zinc-, lead-, and copper-coated steel wire and stainless steel wire exposed in 1936 has been prepared for publication as a "Twenty-Year Atmospheric Corrosion Investigation of Zinc-Coated and Uncoated Wire and Wire Products," *ASTM Special Technical Publication No. 290.*

Committee A-7 on Malleable-Iron Castings:

Data from the first year of exposure of standard malleable, pearlitic, nodular, and heat-treated nodular iron are appended to the 1961 Annual Report of Committee A-7. Specimens are being withdrawn in order to develop the three-year exposure data for this study.

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys:

The seventh inspection of corrosion-resistant steels and architectural and structural applications was completed during the year. A report of this inspection is appended to the 1961 Annual Report of Committee A-10.

One set of the stressed and unstressed stainless steel wire springs exposed at six test sites in 1958 was removed after exposure of one year. A sampling of these specimens was subjected to fatigue failure tests. These data will be published with the three-year exposure data so that any trends of the one-year data may be more definitely established.

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys:

Continuous recording apparatus to measure temperature, time-of-wetness, and sulfur dioxide activity at four U.S. and three Canadian corrosion sites was started in January, 1961. The continuous data recording by this apparatus will be compared with month-to-month exposure corrosion data obtained from adjacent specimens of steel and zinc.

The first-year exposure data from the 1957 program to study the atmospheric corrosion of 77 non-ferrous metals and the 1958 program to study galvanic corrosion of panel-type specimens encompassing aluminum alloys, mild steel, red brass, stainless steel, chromium-plated steel, and monel, in contact with two magnesium alloys are appended to the 1961 Annual Report of Committee B-3.

Committee B-4 on Metallic Materials for Electrical Heating, Electrical Resistance, and Electrical Contacts:

Two louvered shelters containing static contact wires and film foil were installed at Newark (New York area), N. J., and Kure Beach (800-ft), N. C. Each shelter contains 720 wires to make 360 static contacts of gold, silver, nickel, tin, palladium, and beryllium-copper wires, plus a variety of alloys, electroplated coatings, and metallic dips with various treatments. Electrolytic reduction tests are being made on 100 metal foils, including the aforementioned metals and coatings plus brass. The data obtained from the initial resistance measurements at the time of installation have been prepared. Resistance measurements following the first period of exposure will be made in July, 1961.

Committee B-6 on Die-Cast Metals and Alloys:

The 20-yr exposure data for zinc alloy die castings AC43A, AC41A, and AG40A are appended to the 1961 Annual Report of Committee B-6. These data represent the final report for magnesium and zinc alloys exposed in 1939 at five outdoor and three storage sites.

The 20-yr exposure data from three aluminum alloys exposed in 1934 to determine change in tensile strength and elongation during atmospheric exposure and to determine the difference in performance of the high-purity alloys are also appended to the 1961 Annual Report of the committee.

Committee B-7 on Light Metals and Alloys, Cast and Wrought:

The complete 5-yr exposure data for cast and permanent mold castings and wrought aluminum and magnesium alloys exposed in 1953 are appended to the 1960 and 1961 Annual Reports of the committee.

Committee B-8 on Electrodeposited Metallic Coatings:

Program No. 7 is being developed to determine the performance of decorative finishes on zinc-base die castings. This program will include a variety of copper- and chromium-platings on steel and will include various thicknesses of duplex-type nickel coatings.

The one-year inspection of Program No. 5 covering copper-nickel-chromium coatings on steel has been made.

Program No. 4 was designed to determine the effect of nickel strike versus copper strike under chromium and to determine the effect of heavy crack-free chromium, buffing, bright nickel, and bright versus Watts nickel, on the life of electroplated coatings on steel. The final report of this program is appended to the 1961 Annual Report of the committee.

The final report of Program No. 101 on chromate coatings on zinc plating, utilizing electrolytic, olive drab, clear, and iridescent chromate coatings, with untreated control specimens, is appended to the 1961 Annual Report of the committee.

Committee C-19 on Structural Sandwich Constructions:

A fourth atmospheric exposure program of a variety of structural sandwich construction panels as used in the construction industry is being prepared for a new atmospheric corrosion test at State College, Pa., and Kure Beach (80-ft site), N.C.

ATMOSPHERIC EXPOSURE TEST SITES

United States:

The use permit obtained by the Society for the possible construction of a test site two miles west of Tucson, Ariz., has been cancelled.

Canada:

The National Research Council of Canada has under its jurisdiction eight atmospheric exposure test sites. These test sites do not have the capacity to permit their use by the general public; however, there naturally exists an interest as to the performance of materials in these atmospheres. It seemed pertinent therefore that information as to the type and location of the test sites as well as the reports of the National Research Council and ASTM covering exposure data of materials exposed at these test sites be recorded.

TABLE I.—NATIONAL RESEARCH COUNCIL, CANADA, ATMOSPHERIC EXPOSURE SITES.

Rural, Low-Humidity Atmosphere	Saskatoon, Sask. (52°07′N–106°38′W)
Rural, High-Humidity Atmosphere	Ottawa, Ont. (45°25′N–75°42′W)
Rural-Industrial Atmosphere	Trail, B. C. (49°06′N–117°42′W)
Industrial Atmosphere	Montreal, Que. (45°31′N–74°31′W)
Marine-Industrial Atmosphere	Halifax, N. S. (44°39′N–63°36′W)
Marine Atmosphere	York Redoubt, N. S. (44°39′N–63°36′W)
	Rocky Point (Esquimalt), B. C. (48°28′N–123°24′W)
Arctic Atmosphere	Norman Wells, N. W. T. (65°17′N–126°51′W)

A few years ago arrangements were completed between the Division of Building Research of the National Research Council of Canada, and ASTM for the reciprocal use of eight atmospheric exposure test sites in Canada. These sites were selected some 8 years ago by the National Research Council to include six different atmospheric conditions that were considered essential to cover the range of Canadian atmospheres. The six conditions are listed in Table I.

Each of the test sites is equipped with pipe frames 57 ft in length for mounting specimen racks at 30 deg from the horizontal. Auxiliary brackets are also available for 45-deg mounting of specimens. Standard specimen sizes for the exposure racks are: 4 by 6 in. and 6 by 9 in.

The sites at Halifax (2 locations), Ottawa, Saskatoon, and Norman Wells are administered by the staff of the NRC regional laboratories. The sites at Montreal, Esquimalt (Rocky Point), and Trail are supervised, respectively, through the cooperation of the staff of the Research and Development Laboratories, Canadian National Railways; the Pacific National Laboratories, Defense Research Board; and the Research Department, Consolidated Mining and Smelting Co.

Considerable attention has been paid to the development of apparatus to record time-of-wetness and temperature of the specimens as well as to determine the sulfur dioxide atmospheric pollution rate (1,2,3,4). An exposure program utilizing this apparatus has been sponsored by ASTM Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys, Subcommittee VII on Weather, in which time-of-wetness measurements have been made continuously at Cleveland, Ohio, South Bend, Pa., Kure Beach (80- and 800-ft sites), N. C., Ottawa, Ont., Trail, B. C., Halifax, N. S., and Esquimalt (Rocky Point), B. C. Measurements of sulfur dioxide have also been made at the first six sites. It is planned to expose specimens of steel and zinc each month for six months. This will permit a systematic study of the effect of known atmospheres with respect to corrosion of these metals. The measurement of atmospheric chlorides is also under investigation (4).

Two atmospheric exposure test studies were designed by ASTM Committee B-3 to determine the relative corrosivity of the NRC, ASTM, and other sites in terms of mild steel and commercial grade rolled zinc. A complete report of the first study covering 19 sites has been published (5) and is a valuable reference for the selected use of the Canadian sites. The second, more comprehensive, study began in June, 1960, and includes some 39 exposure sites in Canada, England, the United States, the Panama Canal Zone, and the Philippines.

Presently the National Research Council has test studies covering architectural metals at its sites that include a variety of aluminum alloys, copper, lead, magnesium, rolled zinc, various compositions of steels and stainless steels (302, 316, 430), and proprietary alloys. Many types of electrodeposited, sprayed, and chemical protective coatings (6,7) as well as galvanic couples of aluminum alloys riveted to steel, zinc, and copper are also on exposure with some data published (6).

Respectfully submitted on behalf of the committee,

K. G. COMPTON,
Chairman.

S. F. ETRIS,
Secretary.

REFERENCES

(1) P. J. Sereda, "Measurement of Surface Moisture and Sulfur Dioxide Activity at Corrosion Sites," ASTM BULLETIN, No. 246, May, 1960, p. 47 (TP 107).

(2) P. J. Sereda, "Measurement of Surface Moisture—Second Progress Report," ASTM BULLETIN, No. 238, May, 1959, p. 61 (TP 129).

(3) P. J. Sereda, "Measurement of Surface Moisture—A Progress Report," ASTM BULLETIN, No. 228, Feb., 1958, p. 53 (TP 59).

(4) M. R. Foran, E. V. Gibbons, and J. R. Wellington, "Measurement of Atmospheric Sulphur Dioxide and Chlorides," *Chemistry in Canada*, Vol. 10, No. 5, May, 1958.

(5) Report of Subgroup of Subcommittee VII, on Corrosiveness of Various Atmospheric Test Sites as Measured by Specimens of Steel and Zinc, *Proceedings*, Am. Soc. Testing Mats., Vol. 59, p. 183 (1959).

(6) The Corrosion Behavior of the Major Architectural and Structural Metals in Canadian Atmospheres. Summary of two-year results. Associate Committee on Corrosion Research and Prevention, National Research Council (Canada), Feb., 1959.

(7) J. Harris, "The Results of Exposure of Series I Painted Panels," prepared for the Associate Committee on Corrosion Research and Prevention (In Press).

REPORT OF COMMITTEE C-1
ON
CEMENT*

Committee C-1 on Cement held two meetings during the year: in Atlantic City, N. J., on July 1, 1960, and in Denver, Colo., on Dec. 6, 1960.

The committee records with sorrow the deaths of three of its members: H. F. Clemmer, who joined in 1940; J. R. Dwyer and P. J. Freeman, Honorary Members, both of whom joined in 1920. Mr. Dwyer served as Technical Assistant to Committee C-1 from 1931 to 1955.

At the 1960 Annual Meeting of the Society, C. H. Scholer received the ASTM Award of Merit.

In recognition of their long association with Committee C-1, and their many valuable contributions to its work, W. H. Klein, W. C. Hanna, and M. A. Swayze were made Honorary Members of the committee.

NEW TENTATIVE

The committee recommends for publication as tentative the Specifications for Processing Additions for Use in the Manufacture of Portland Cement as appended hereto.[1]

REVISION OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Methods of Chemical Analysis of Portland Cement (C 114 - 58 T):[2]

Section 1(b).—Change to read as follows:

	Section
Vinsol Resin (Methoxyl Method) . .	3 to 5
Ignition Loss of Portland Blast-Furnace Slag Cement and Slag Cement.	6

Section 6.—Delete the present Sections 6 to 29, renumbering the present Section 30 as Section 6.

Tentative Methods of Sampling Hydraulic Cement (C 183 - 60 T):[3]

Section 1.—Delete the word "the."

Section 2.—Change to read as follows under the revised heading "Kinds, Number, and Size of Samples: By Whom Taken:"

2. (*a*) A cement sample secured in one operation shall be termed a "grab sample." A sample obtained by means of an automatic sampling device that continuously samples a cement stream shall be termed a "continuous sample." Where a grab sample represents a cement stream for a brief period, a continuous sample may represent the cement stream for short or extended periods of time, as desired by the sampling agency.

(*b*) Grab samples taken at prescribed intervals over a period of time may be combined to form a "composite sample" representative of the cement produced during that period of time. A series of individual continuous samples secured at prescribed intervals may also be combined to form a composite sample representative of the period in question. A single continuous sample secured by means of an automatic sampling device continuously over the period in question is also a composite sample for the period.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] The new tentative appears in the 1961 Book of ASTM Standards, Part 4.

[2] 1958 Book of ASTM Standards, Part 4.

[3] 1960 Supplement to Book of ASTM Standards, Part 4.

(*c*) Samples on which physical or chemical tests are to be made shall be called "test samples." Test samples shall be either grab samples or composite samples, as may be specified, and one test sample shall represent not more than 2000 barrels unless otherwise specified by the purchaser.

(*d*) Individual samples to be composited shall weigh at least 5 lb. Individual test samples on which all specified tests are to be made shall weigh at least 10 lb.

(*e*) The sampling shall be done by or under the direction of a responsible representative of the purchaser.

(*f*) Samples shall be packed by the purchaser in moisture-proof, airtight containers that are to be numbered consecutively in the order in which the samples are taken. The containers shall be crated, if necessary, by the manufacturer and shipped at the expense of the purchaser.

Section 3(a).—Change to read as follows:

(*a*) *From the Conveyor Delivering to Bulk Storage.*—One test sample of 5 lb or more shall be taken from at least each 1000 barrels passing over the conveyor, except that the sample shall represent not more than 5 to 6 hr of cement production (conveyor operation). The sample may be secured by either the grab or the composite method. When obtaining a composite test sample, it shall be secured by combining approximately equal weights of the cement taken at regular intervals, each portion representing not more than 200 barrels.

Section 3(f).—Delete the next to the last sentence.

Section 4(a).—Delete the first sentence.

Section 4(b).—In the first two sentences, change the expression "test samples" to "individual samples."

Tentative Specifications for Portland Blast-Furnace Slag Cement (C 205 – 60 T):[3]

Table I.—Delete the phrase "Manganic oxide (Mn_2O_3), max, per cent . . . 1.5."

Note 1.—Delete the entire note, renumbering Note 2 as Note 1.

Tentative Specifications for Air-Entraining Additions for Use in the Manufacture of Air-Entraining Portland Cement (C 226 – 58 T):[2]

Section 3(c).—In the last line of the second paragraph change the term "per cent" to "percentage points."

Section 10(c).—Change to read as follows:

(*c*) *Resistance to Freezing and Thawing.*—Method of Test for Resistance of Concrete Specimens to Rapid Freezing and Thawing in Water (ASTM Designation: C 290), or Method of Test for Resistance of Concrete Specimens to Slow Freezing and Thawing in Water or Brine (ASTM Designation: C 292) shall be used, except that the specimens shall be tested at the end of the 28-day curing period. In case of a disagreement between the results obtained by these two methods, the results obtained by Method C 290 shall govern.

Tentative Specifications for Flow Table for Use in Tests of Hydraulic Cement (C 230 – 57 T):[2]

Section 5(a).—Add the following new sentence at the end of this paragraph:

A circular shield approximately 10 in. in diameter, with a center opening approximately 4 in. in diameter, made of nonabsorbing material not attacked by the cement, shall be used with the flow mold to prevent mortar from spilling on the table top.

Tentative Method of Test for Flexural Strength of Hydraulic Cement Mortars (C 348 – 57 T):[2]

Section 2(h).—Add the following new Notes 2 and 3 at the end of this paragraph:

NOTE 2.—Most hydraulic compression machines designed for breaking 2-in. cubes have a relatively small diameter lower bearing surface directly centered below the upper spherically seated head, on which close-fitting pedestals of appropriate heights are set for breaking 2-in. cubes and 2 by 4-in. or 3 by 6-in. cylinders. The base plate of the flexure testing apparatus shown in Fig. 3 is designed to rest on the low pedestal intended for tests on 3 by 6-in. cylinders.

NOTE 3.—In the absence of self-centering arrangements on machines with large lower bearing surfaces, the center of this surface directly below the center of the upper spherically seated head shall be accurately located. A circle or concentric circles of appropriate diameters shall be scribed on the lower bearing surface around this point. A cylindrical pedestal of appropriate diameter and height shall be obtained. End faces of the pedestal must be plane and parallel and at 90 deg to the axis of the cylinder. The upper face shall have a diameter of 3.05 in.

Section 2(i).—Add the following new Note 4 at the end of this paragraph:

NOTE 4.—A number of types of briquet testing machines have insufficient space between the hangers for the briquet clips and the frame of the machine to admit the wider assembly needed for flexure tests on prisms, as shown in Fig. 4. Laboratories intending to convert briquet machines for this use should check this dimension before purchase of the adapting equipment.

Section 6.—In the last sentence, change the phrase "Notes 2 and 3," to read "Notes 5 and 6," and renumber the notes which follow this section accordingly.

Section 11(a).—Change the heading preceding this section to read "Testing Procedure." In the second sentence replace the words "plastic cloth" with the words "waterproof plastic." At the beginning of the third sentence add the words "For older specimens,". Add the following new Note 7 after Paragraph (*a*):

NOTE 7.—The flexural strength of prisms is quickly affected by drying which produces skin tension in the specimens and yields low strengths. Specimens for test at 24 hr should, therefore, be removed from molds preferably at that age and tested immediately. If there is a delay in testing, specimens shall be wrapped in waterproof plastic and placed in the moist closet until immediately before testing. After breaking, rewrap in plastic until compressive tests are made.

Section 11(b).—Add a new sentence at the end of this section to read: "Cloths used for wiping 24-hr specimens shall not be more than faintly damp."

Section 11(c).—Change to read as follows:

(*c*) Center the pedestal on the base plate of the machine directly below the center of the upper spherical head, and place the bearing plate and support edge assembly on the pedestal. Attach the center-loading device to the spherical head. Turn the specimen on its side with respect to its position as molded and place it on the supports of the testing device. The longitudinal center line of the specimen shall be set directly above the mid-point of both supports. Adjust the center-point loading device so that its bearing edge is at exactly right angles to the length of the prism and parallel to its top face as placed, with the center of the bearing edge directly above the center line of the prism and at the center of the span length. Care shall be exercised to insure that contact between the specimen and loading edge is continuous when the load is applied. The load shall be applied at the rate of 600 ± 25 lb per min and shall be indicated within an accuracy of ± 1 per cent on a dial graduated in increments of not more than 10 lb. Total maximum load shall be estimated to the closest 5 lb.

Figure 3.—Revise the principal caption to read "Special Components for Adapting Compression Testing Machine for Testing 40 by 40 by 160-mm Prisms." Revise the caption for Fig. 3(*a*) to read "Base Plate and Support Edges." Revise the caption for Fig. 3(*b*) to read "Center-Point Loading Component." Substitute the new Fig. 3(*b*) as shown in the accompanying Fig. 1.[4]

Tentative Method of Test for False Set of Portland Cement (Mortar Method) (C 359 – 56 T):[2]

New Section 2.—Add the following new Section 2 under the heading "Definitions," renumbering the subsequent sections accordingly:

2. (*a*) *False Set*.—False set is the rapid development of rigidity in a mixed portland

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[4] The new figure is not included in this report but appears in the revised method of test; see 1961 Book of ASTM Standards, Part 4.

cement paste, mortar, or concrete without the evolution of much heat, which rigidity can be dispelled and plasticity regained by further mixing without addition of water. Grab set, premature stiffening, hesitation set, early stiffening, and rubber set are terms referring to the same phenomenon, but false set is the preferred term.

(*b*) *Quick or Flash Set.*—Quick or flash set is the rapid development of rigidity in a mixed portland cement paste, mortar, or concrete, usually with the evolution of considerable heat, which rigidity cannot be dispelled nor can the plasticity be regained by further mixing without addition of water. Quick set is the preferred term.

Section 4(d).—Change the second sentence of the second paragraph to read: "In a similar manner, determine, after moving the Vicat apparatus to the desired location, the penetrations at intervals of 5, 8, and 11 min after the beginning of the mixing." After this sentence add a new sentence to read: "The filled container is not to be moved until these measurements are completed."

New Section 7.—Add the following new Section 7 under the heading "Interpretation":

7. False set is generally conceded to result from the rapid crystallization of gypsum from the solution of dissolved calcium sulfate hemihydrate or soluble anhydrite occurring when water is added to the cement. Heat generated during finish grinding of the cement clinker almost inevitably results in conversion of some of the gypsum added for control of setting, either or both to hemihydrate or soluble anhydrite.

From a placing and handling standpoint, false set tendencies in cement will cause no difficulties where concrete is mixed for a longer time than usual, such as usually occurs in transit mixing, or where it is remixed prior tc placing or transporting, such as occurs in concrete pumping operations. It is most apt to be noticeable where concrete is mixed for a short period of time in stationary mixers and transported to the forms in non-agitating equipment, such as on some paving jobs.

False set per se has no deleterious effects on the quality of concrete; however, cements with false setting tendencies usually require slightly more mixing water to produce the same consistency, which may be expected to result in slightly lower strengths and increased drying shrinkage.

TENTATIVE REVISION OF STANDARD

The committee recommends tentative revision as follows of the Standard Specifications for Portland Cement (**C 150 – 60**):[3]

Section 3.—Change to read as follows:

3. For the purpose of these specifications, portland cement is the product obtained by pulverizing clinker consisting essentially of hydraulic calcium silicates, to which no additions have been made subsequent to calcination other than water and/or untreated calcium sulfate, except that additions of other materials not to exceed 1.0 per cent may be interground with the clinker at the option of the manufacturer, provided such materials are added in amounts not to exceed the amount that has been shown to be non-harmful by tests prescribed in the Specifications for Processing Additions for Use in the Manufacture of Portland Cement (ASTM Designation: C 465).

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee recommends that the tentative revision of Section 4 of both the Standard Specifications for Portland Cement (**C 150 – 60**)[3] and the Standard Specifications for Air-Entraining Portland Cement (**C 175 – 60**)[3] be approved for reference to letter ballot of the Society for adoption as standard.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Methods of Chemical Analysis of Portland Cement (C 114 – 58):[2]

In order to make use of improved testing procedures, the following additions and deletions, accompanied by all necessary editorial revisions of cross-references between sections, are recommended:

Section 1(b).—Retain the first part of this section in its present form and change the latter part to read as follows:

	Section
Titanium Dioxide	24 to 26
Phosphorus Pentoxide	27 and 28
Manganic Oxide	29 and 30
Insoluble Residue	31
Chloroform Soluble Organic Substances	32 and 33
Free Calcium Oxide	34 to 40
OPTIONAL METHODS	
Silicon Dioxide	41
Calcium Oxide	42 and 43
Magnesium Oxide:	
Method A—Gravimetric	44
Method B—Alkalimetric	45 and 46
Method C—8-Hydroxyquinoline	47 and 48
Sulfur Trioxide	49 to 52
Sodium and Potassium Oxide:	
Method A	53 and 54
Method B	55 to 58
Titanium Dioxide:	
Method A	59 to 61
Method B	62 to 64
Phosphorus Pentoxide	65 and 66
Manganic Oxide	67 and 68
Free Calcium Oxide	69 to 72

Sections 24 to 30.—Under the heading "Titanium Dioxide" insert Sections 9 to 11 of C 114 - 58 T as Sections 24 to 26, renumbering the present Sections 24 to 30 as Sections 27 to 33.

Sections 31 and 32.—Delete the present Sections 31 and 32 under the heading "Free Calcium Oxide in Portland Cement and Clinker" and insert Sections 19 to 25 of C 114 - 58 T as Sections 34 to 40 under this heading.

Sections 33 to 44.—Renumber the present Sections 33 to 44 as Sections 41 to 52.

Sections 45 and 46.—Renumber the present Sections 45 and 46 as Sections 53 and 54 under the new heading "Sodium Oxide and Potassium Oxide—*Optional Method A.*" Then, under the new heading "*Optional Method B—Determination by Flame Photometry, Using the Direct-Intensity Method,*" insert Sections 15 to 18 of C 114 - 58 T as Sections 55 to 58.

New Sections 59 to 64.—Under the heading "Titanium Dioxide—*Optional Method A—Peroxide Method Using Nessler Tubes*" insert Sections 12 to 14 of C 114 - 58 T as Sections 59 to 61. Under the heading "*Optional Method B—Titration with Methylene Blue*" insert Sections 6 to 8 of C 114 - 58 T as Sections 62 to 64.

Sections 47 to 50.—Renumber the present Sections 47 to 50 as Sections 65 to 68.

Sections 51 and 52.—Delete the present Sections 51 and 52 under the heading "Free Calcium Oxide—*Optional Method*" and insert Sections 26 to 29 of C 114 - 58 T as Sections 69 to 72.

Section 53.—Renumber the present Section 53 as Section 73, and revise the subsection entitled "*Minor Components*" by inserting "TiO_2 . . . Titanium dioxide" as the third item in the listing.

Standard Specifications for Portland Cement (C 150 - 60):[3]

Section 3, Footnote 3.—Add the following new Paragraph (*c*) to Footnote 3:

(*c*) The material known commercially as Polyfon T (a highly purified water-soluble sodium lignosulfonate, made from lignin produced by the alkaline process for producing paper pulp) manufactured by the West Virginia Pulp and Paper Co., when added in an amount not exceeding 0.05 per cent of the clinker weight in the finished grinding of portland cement may be used.

Table I.—Add the phrase "Sum of tricalcium silicate and tricalcium aluminate, max, per cent . . ." to the first column, and opposite that in the column headed "Type II" insert the expression "58[e]."

Replace the present Footnote *c* with the following:

[e] The expressing of chemical limitations by means of calculated assumed compounds does

not necessarily mean that the oxides are actually or entirely present as such compounds.

When the ratio of percentages of aluminum oxide to ferric oxide is 0.64 or more, the percentages of tricalcium silicate, dicalcium silicate, tricalcium aluminate and tetracalcium aluminoferrite shall be calculated from the chemical analysis as follows:

Tricalcium silicate = (4.071 × per cent CaO) − (7.604 × per cent SiO_2) − (6.717 × per cent Al_2O_3) − (1.429 × per cent Fe_2O_3) − (2.852 × per cent SO_3)

Dicalcium silicate = (2.868 × per cent SiO_2) − (0.7544 × per cent C_3S)

Tricalcium aluminate = (2.650 × per cent Al_2O_3) − (1.691 × per cent Fe_2O_3)

Tetracalcium aluminoferrite = 3.043 × per cent Fe_2O_3

When the alumina-ferric oxide ratio is less than 0.64, a calcium aluminoferrite solid solution (expressed as ss ($C_4AF + C_2F$) is formed. Contents of this solid solution and of tricalcium silicate shall be calculated by the following formulas:

ss ($C_4AF + C_2F$) = (2.100 × per cent Al_2O_3) + (1.702 × per cent Fe_2O_3)

Tricalcium silicate = (4.071 × per cent CaO) − (7.604 × per cent SiO_2) − (4.478 × per cent Al_2O_3) − (2.859 × per cent Fe_2O_3) − (2.852 × per cent SO_3).

No tricalcium aluminate will be present in cements of this composition. Dicalcium silicate shall be calculated as previously shown.

In the calculation of C_3A, the values of Al_2O_3 and Fe_2O_3 determined to the nearest 0.01 per cent shall be used. In the calculation of other compounds the oxides determined to the nearest 0.1 per cent shall be used.

Values for C_3A and for the sum of $C_4AF + 2C_3A$ shall be reported to the nearest 0.1 per cent. Values for other compounds shall be reported to the nearest 1 per cent.

Add the following new Footnote *e*:

[e] This limit applies when moderate heat of hydration is required and tests for heat of hydration are not requested.

Table II.—Opposite the phrase "Soundness: Autoclave expansion, max, per cent . . ." change the value "0.50" to "0.80."

Standard Specifications for Air-Entraining Portland Cement (C 175 – 60):[3]

Table I.—Add the phrase "Sum of tricalcium silicate and tricalcium aluminate, max, per cent . . ." to the first column, and opposite that in the column headed Type IIA insert the expression "58[c]."

Replace the present footnote "*a*" with wording corresponding to that for the proposed new Footnote "*c*" for C 150.

Add a new Footnote *c* with wording corresponding to that for the proposed new Footnote *e* for C 150 as shown above.

Table II.—Opposite the phrase "Soundness: Autoclave expansion, max, per cent . . ." change the value "0.50" to "0.80."

STANDARD CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Standard Specifications for Natural Cement **(C 10 – 54)**.[2]

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Specifications for:

Portland-Pozzolan Cement **(C 340 – 58 T)**.[2]

Tentative Methods of Test for:

Bleeding of Cement Pastes and Mortars **(C 243 – 58 T)**,[2]

Calcium Sulfate in Hydrated Portland Cement Mortar **(C 265 – 58 T)**,[2]

Time of Setting of Hydraulic Cement by Gillmore Needles **(C 266 – 58 T)**,[2]

Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency **(C 305 – 59 T)**,[5] and

Compressive Strength of Hydraulic Cement Mortars (Using Portions of Prisms Broken in Flexure) **(C 349 – 59 T)**.[5]

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following standards and tentatives:

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[5] 1959 Supplement to Book of ASTM Standards, Part 4.

Standard Specifications for Masonry Cement (C 91 – 60):[3]

Section 30(c).—Change to read as follows:

(*c*) *Calculations.*—Calculate the percentage of water soluble alkali as Na_2O to the nearest 0.01 as follows:

$$\text{Per cent soluble } Na_2O = \frac{\text{ppm } Na_2O \text{ in solution in 100-ml flask}}{1500}$$

$$\text{Per cent soluble } K_2O = \frac{\text{ppm } K_2O \text{ in solution in 100-ml flask}}{1500}.$$

Then, per cent $K_2O \times 0.658 =$ per cent Na_2O equivalent of K_2O, and per cent $Na_2O +$ per cent Na_2O equivalent of $K_2O =$ total soluble alkali calculated as Na_2O.

Standard Method of Test for Compressive Strength of Hydraulic Cement Mortars (Using 2-In. Cube Specimens) (C 109 – 58):[2]

Section 8(b).—In the first sentence, change "Section 5" to "Section 6."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[6]

This report has been submitted to letter ballot of the committee, which consists of 84 voting members; 69 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. R. LITEHISER,
Chairman.

W. J. McCOY,
Secretary.

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[6] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-2
ON
MAGNESIUM OXYCHLORIDE AND MAGNESIUM OXYSULFATE CEMENTS*

Committee C-2 on Magnesium Oxychloride and Magnesium Oxysulfate Cements held no meetings during the year. A conference of officers and members of the committee was held on October 13, 1960, in Washington D.C. It was the decision at this conference to request that the committee be placed on an inactive status, pending further developments in this field. At its meeting on January 17, 1961, the Board of Directors placed the committee on inactive status and appointed J. B. James, Dow Chemical Co., as Acting Chairman. All secretarial matters will be handled at ASTM Headquarters.

A Subcommittee on Review was appointed with the responsibility of reviewing all existing standards under the jurisdiction of the committee. The subcommittee membership consists of: J. B. James, Dow Chemical Co.; N. V. Becker, Johns-Manville Corp.; W. H. Kibbel, Jr., Food Machinery and Chemical Corp.; C. C. Martin, The Standard Magnesia Co.; Frank Jaros, Allied Compositions Co. Inc.; and a representative from the Bureau of Ships.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Specification for Oxychloride Magnesia (**C 275 – 56 T**) be approved for reference to letter ballot of the Society for adoption as standard without revision.

REAPPROVAL OF STANDARDS

The committee recommends reapproval of the following standards which have stood for six or more years without revision:

Standard Specifications for:

Magnesium Chloride (**C 276 – 53**), and
Magnesium Sulfate, Technical Grade (**C 277 – 53**).

Standard Methods of Test for:

Bulk Density of Magnesium Oxychloride Cements (**C 248 – 52**),
Chemical Analysis of Magnesium Chloride (**C 245 – 52**),
Chemical Analysis of Magnesium Sulfate, Technical Grade (**C 244 – 52**),
Compressive Strength of Magnesium Oxychloride Cements (**C 257 –52**),
Consistency of Magnesium Oxychloride Cements by the Flow Table (**C 255 – 52**),
Flexural Strength of Magnesium Oxychloride Cements (Using Simple Bar with Two-Point or Single-Point Loading) (**C 256 – 52**),
Ignition Loss and Active Calcium Oxide in Magnesium Oxide for Use in Magnesium Oxychloride Cements (**C 247 – 57**),
Linear Change of Magnesium Oxychloride Cements (**C 253 – 52**),
Linear Contraction of Magnesium Oxychloride Cements (**C 252 – 52**),
Mixing Magnesium Oxychloride Cement Compositions with Gauging Solution (for Preparation of Specimens for Laboratory Tests) (**C 251 – 52**),
Physical Testing of Magnesia for Magnesium Oxychloride Cements (**C 246 – 52**),

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Sampling Magnesium Oxychloride Compositions and Ingredients (**C 237 – 51**),
Setting Time of Magnesium Oxychloride Cements (**C 254 - 52**),
Sieve Analysis of Magnesium Oxychloride Compositions, Aggregates, and Fillers (**C 238 – 51**),
Sieve Analysis of Plastic Calcined Magnesia (**C 239 – 51**),
Slump Test for Field Consistency of Magnesium Oxychloride Cements (**C 249 – 52**).

Standard Specification and Method for:

Field Determination of Specific Gravity of Gauging Solutions for Magnesium Oxychloride Cements (**C 250 – 52**).

This report has been submitted to letter ballot of the Subcommittee on Review which consists of 6 members, all of whom voted affirmatively.

Respectfully submitted on behalf of the committee,

J. B. JAMES,
Acting Chairman.

REPORT OF COMMITTEE C-3
ON
CHEMICAL-RESISTANT MORTARS*

Committee C-3 on Chemical-Resistant Mortars held two meetings during the year: on Oct. 20 and 21, 1960, in Suffern, N. Y., and on Feb. 23 and 24 1961, in Washington, D. C.

The committee consists of 18 members of whom 7 are classified as producers, 7 as consumers, and 4 as general interest members, with 2 consulting members.

The committee approved a change in title and scope of the committee which has been submitted to the Board of Directors. The change will expand the coverage to materials other than mortars that are in the category of chemical-resistant nonmetallic materials used primarily for protection against corrosive chemicals.

NEW TENTATIVE

The committee recommends for publication as tentative the proposed Specification for Chemically Setting Silicate-Type Chemical-Resistant Mortars, as appended hereto.[1]

REVISION OF TENTATIVE

The committee recommends revisions as follows of the Tentative Methods of Test for Working and Initial Setting Times of Chemically Setting, Silicate-Type Chemical-Resistant Mortars (**C 414 – 58 T**)[2] and the continuation of the method as tentative:

Section 4.—Add a new Paragraph (*a*) to read as follows, relettering the present Section 4 as Paragraph *b*:

(*a*) All material used in this test method shall be stored for at least 16 hr prior to use at the standard test temperature as described in Paragraph (*b*).

Section 5(*a*).—Delete the third sentence.

Section 6.—Change the first sentence to read as follows:

Remove approximately 15-g portions of the silicate mortar at 5 min (maximum) intervals, and trowel on the horizontal surface of a clean dry brick or asbestos-cement board.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Method of Tests for Tensile Strength of Resin-Type Chemical-Resistant Mortars (C 307 – 55):[2]

Section 8.—Change the second sentence to read as follows:

The specimens shall then be aged for a period of at least six additional days at 23 ± 2.2 C (73.4 ± 4 F).

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[1] The new tentative appears in the 1961 Book of ASTM Standards, Part 4.

[2] 1958 Book of ASTM Standards, Part 4.

Section 9(b).—Change the second sentence to read as follows:

> They shall be pulled at a rate of 6000 ± 500 psi per min, or at a rate of 0.20 to 0.25 in. per min of crosshead movement (speed of movement when the machine is running without a load).

Standard Methods of Test for Working and Setting Times of Resin-Type Chemical-Resistant Mortars (C 308 – 55):[2]

Section 1.—Delete the second sentence of the scope.

Section 4.—Add a new Paragraph *a* to read as follows relettering the present Section 4 as Paragraph *b*:

> (*a*) All materials used in this test method shall be stored for at least 16 hr prior to use at the standard test temperature as described in Paragraph (*b*).

Section 5(a).—Delete the third sentence.

Section 6.—Change the first sentence to read as follows:

> Remove approximately 15-g portions of the resin mortar at specific intervals and trowel on the horizontal surface of a clean dry brick or asbestos-cement board.

Section 7(a).—Change the first sentence to read as follows:

> Immediately after mixing is complete, approximately 50 g of the mortar shall be removed with a trowel and buttered on the edges of two clean dry bricks.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Method of Test for:

Absorption and Apparent Porosity of Chemical-Resistant Mortars (**C 413 – 58 T**),[2] and
Chemical Resistance of Mortars (**C 267 – 59 T**).[3]

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following standards and tentatives:

Recommended Practice for Use of Sulfur-Type Chemical-Resistant Mortars (C 386 – 60):[4]

Section 9.—Add a sentence at end of the section to read as follows:

> The chemical resistance of this mortar may be determined by the Method of Test for Chemical Resistance of Mortars (ASTM Designation: C 267).

Recommended Practice for Use of Chemically Setting Silicate-Type Chemical-Resistant Mortars (C 397 – 60):[3]

Section 8.—Add a new sentence at the end of the section to read as indicated above for Section 9 of Recommended Practice C 386.

Tentative Recommended Practice for Use of Hydraulic Cement Mortars in Chemical-Resistant Masonry (C 398 – 60 T):[4]

Section 15.—Add a new sentence at the end of the section to read as indicated above for Section 9 of Recommended Practice C 386.

Recommended Practice for Use of Resin-Type Chemical-Resistant Mortars (C 399 – 60):[3]

Section 11.—Add a new sentence at the end of the section to read as indicated above for Section 9 of Recommended Practice C 386.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

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[3] 1959 Supplement to Book of ASTM Standards, Part 4.

[4] 1960 Supplement to Book of ASTM Standards, Part 4.

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

This report has been submitted to letter ballot of the committee, which consists of 18 members; 18 members returned their ballots, all of whom voted affirmatively.

Respectfully submitted on behalf of the committee,

J. R. ALLEN,
Chairman.

E. A. REINECK,
Secretary.

REPORT OF COMMITTEE C-4
ON
CLAY PIPE*

Committee C-4 on Clay Pipe held one meeting during the year: in Atlantic City, N. J., on June 28, 1960, during the Annual Meeting of the Society.

The committee consists of 29 members of whom 12 are classified as producers, 14 as consumers, and 3 as general interest members.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1960 Annual Meeting, Committee C-4 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specifications for:

Standard Strength Ceramic Glazed Clay Pipe (C 462 - 60 T), and
Extra Strength Ceramic Glazed Clay Pipe (C 463 - 60 T).

Revision of Tentative Specifications for:

Standard Strength Ceramic Glazed or Unglazed Clay Sewer Pipe (C 261 - 59 T), with a change in title to read, "Specifications for Standard Strength Unglazed Clay Pipe."
Extra Strength Ceramic Glazed or Unglazed Clay Pipe (C 278 - 57 T), with a change in title to read, "Specifications for Extra Strength Unglazed Clay Pipe."
Vitrified Clay Pipe Joints Using Materials Having Resilient Properties (C 425 - 58 T).

Revision and Reversion to Tentative of Standard Methods of Testing:

Clay Pipe (C 301 - 54).

These recommendations were accepted by the Standards Committee on September 29, 1960, and the new and revised tentative specifications and methods appear in the 1960 Supplement to the Book of ASTM Standards, Part 5.

STANDARD CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Standard Specifications for Clay Flue Linings (**C 315 - 56**).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Specifications for:

Standard Strength Clay Sewer Pipe (**C 13 - 57 T**),
Extra Strength Clay Pipe (**C 200 - 59 T**), and
Standard Strength Perforated Clay Pipe (**C 211 - 57 T**).

Tentative Recommended Practices for:

Installing Vitrified Clay Sewer Pipe (**C 12 - 58 T**).

This report has been submitted to letter ballot of the committee, which consists of 29 members; 24 members returned their ballots, of whom 23 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

C. R. VELZY,
Chairman.

ROBERT G. SCOTT,
Secretary.

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EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-4 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specification for:

Vitrified Clay Liner Plates (C 479 – 61 T).

Revision of Tentative Specification for:

Standard Strength Perforated Clay Pipe (C 211 – 57 T), with a change in title to read, "Tentative Specifications for Standard and Extra Strength Perforated Clay Pipe."

These recommendations were accepted by the Standards Committee on September 12, 1961, and the new and revised tentative specifications appear in the 1961 Book of ASTM Standards, Part 5.

REPORT OF COMMITTEE C-7

ON

LIME*

Committee C-7 on Lime held two meetings during the year: on June 29, 1960, in Atlantic City, N. J., during the Annual Meeting of the Society, and on February 2, 1961, in Cincinnati, Ohio, during ASTM Committee Week.

The committee consists of 51 members, of whom 24 are classified as producers, 8 as consumers, and 16 as general interest members, with 3 honorary members.

Due to the death of its chairman, James A. Murray, Committee C-7 held a special election which resulted in the election of the following officers: H. F. Kriege, chairman; R. K. Thomas and E. T. Carlson, vice-chairmen; and L. E. Johnson, secretary.

The committee regrets the loss of Mr. Murray, who died March 7, 1960, and also Walter C. Voss, an honorary member, who died November 2, 1960.

Walter Walker has been appointed chairman of Subcommittee VIII on Nomenclature, replacing B. M. O'Kelly, who resigned.

The formation of Subcommittee XII on Limestone for the Chemical and Process Industries was cancelled. This product will be handled by Subcommittee III on Lime for the Chemical Industries.

Committee C-7 elected representatives as follows to Committee E-1 on Methods of Testing: R. E. Halsted to Subcommittee 5 on Compression Testing; V. S. Tadsen to Subcommittee 10 on Sieves; W. J. Barrett to Subcommittee 11 on Sub-Sieve Testing; and R. B. Vogel to Subcommittee 12 on Methods for Density. R. K. Thomas was elected as representative to Committee E-15 on Analysis and Testing of Industrial Chemicals. The committee discontinued its representation on Subcommittee 8 on Flexural Strength Test, Subcommittee 9 on Rheological Properties, and Subcommittee 14 on Conditioning and Weathering of Committee E-1.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revision as follows of the Standard Specifications for Hydraulic Lime for Structural Purposes (**C 141 – 55**),[1] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revision may be referred to letter ballot of the Society.

Section 1(b).—Delete the phrase "and as sole cementitious material in concrete."

Section 2.—Change the first line of the Note to read "The purchaser may increase the hydraulicity . . ."

Delete Paragraph (*c*).

Section 3.—In the table, delete the

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[1] 1958 Book of ASTM Standards, Part 4.

headings "Type A" and "Type B," and delete both columns under Type B.

Section 4.—Delete the phrase "For both types A and B hydraulic hydrated lime."

Section 5.—Delete the phrase "For type A hydraulic hydrated lime."

Section 6.—Delete the phrase "For both types A and B hydraulic hydrated lime."

Section 7.—Delete "(*a*) *Type A*"; delete Paragraph (*b*).

Section 15.—Replace with the following new paragraph under the heading "Temperature and Humidity":

> 15. The temperature of the air in the vicinity of the mixing slab and the dry materials, molds, base plates, and mixing bowl, shall be maintained between 20 and 27.5 C (68 and 81.5 F). The temperature of the mixing water, moist closet or moist room, and water in the storage tank shall not vary from 23 C (73.4 F) by more than ±1.7 C (3 F). The moist closet or moist room shall be so constructed as to provide storage facilities for test specimens at a relative humidity of not less than 90 per cent.

Section 20.—Change the reference to read "in accordance with the Method of Test for Time of Setting of Hydraulic Cements by Gillmore Needles (ASTM Designation: C 266)."

Section 25.—In line 5 put a comma after the word "mortar."

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Standard Specifications for:

Normal Finishing Hydrated Lime (**C 6 – 49**),
Quicklime and Hydrated Lime for Cooking of Rags in Paper Manufacture (**C 45 – 25**),
Quicklime for Sulfite Pulp Manufacture (**C 46 – 27**),
Special Finishing Hydrated Lime (**C 206 – 49**),
Hydrated Lime for Masonry Purposes (**C 207 – 49**),
Quicklime for Calcium Carbide Manufacture (**C 258 – 52**), and
Hydrated Lime for Grease Manufacture (**C 259 – 52**).

Standards Definitions of Terms Relating to:

Lime (**C 51 – 47**).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Specifications for:

Quicklime and Hydrated Lime for Water Treatment (**C 53 – 52 T**),
Fly Ash for Use as a Pozzolanic Material with Lime (**C 379 – 56 T**),
Quicklime and Hydrated Lime for Sand-Lime Products (**C 415 – 58 T**),
Pozzolans for Use with Lime (**C 432 – 59 T**), and
Quicklime and Hydrated Lime for Hypochlorite Bleach Manufacture (**C 433 – 59 T**).

EDITORIAL CHANGE

The committee recommends an editorial change as follows in the Tentative Method of Testing Quicklime and Hydrated Lime for Neutralization of Waste Acid (**C 400 – 57 T**):[1]

Section 6(a)(1).—Delete the phrase "in 30 sec" in line 12.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

This report has been submitted to letter ballot of the committee, which consists of 51 members; 36 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. F. KRIEGE,
Chairman.

L. E. JOHNSON,
Secretary.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-8 ON REFRACTORIES*

Committee C-8 on Refractories held two meetings during the year: the ninety-eighth at Bedford Springs, Pa., on Oct. 6, 1960; and the ninety-ninth at Cincinnati, Ohio on Jan. 31, 1961.

The committee consists of 48 members, of whom 26 are classified as producers, 17 as consumers, and 5 as general interest members.

New Tentatives

The committee recommends for publication as tentative the following classifications as appended hereto:[1]

Classifications of:

Mullite Refractories, and
Refractory Granular Dolomite.

Revisions of Tentatives

The committee recommends revisions as appended hereto[2] of the following tentatives and their continuation as tentative:

Tentative Methods of Test for:

Modulus of Rupture of Castable Refractories (C 268 – 55 T), and
Permanent Linear Change on Firing of Castable Refractories (C 269 – 57 T).

Tentative Specifications for:

Steel Pouring Pit Refractories (C 435 – 59 T).

Tentative Revisions of Standards

The committee recommends tentative revisions as indicated of the following standards:

Classification of Fireclay and High-Alumina Refractory Brick (C 27 – 60):[3]

Section 2(b).—Add the following new classes: "85 per cent alumina, 90 per cent alumina, and 99 per cent alumina."

Table I.—Add requirements for the new classes as follows:

High-Alumina Brick	Other Test Requirements
85 per cent alumina....	Alumina content, 85 ± 2.0 per cent
90 per cent alumina....	Alumina content, 90 ± 2.0 per cent
99 per cent alumina....	Alumina content, minimum of 97 per cent

Method for Basic Procedure in Panel Spalling Test for Refractory Brick (C 38 – 58):[3]

Section 4.—Change the fourth sentence to read as follows:

The 9 by 4½-in. and 4½ by 2½-in. or 3-in. faces of all test brick on the panel shall be first coated with primer coating, allowed to dry, and then laid up with refractory kaolin having a PCE value of not less than Cone 34.

Add the following two notes to Section 4:

Note 1.—Primer Coat Composition and Preparation:

Minus 60-mesh, pure, sintered or fused alumina (similar in grade to T-61 tabular alumina)...... 53.0 per cent
Raw white starch (household

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] The new tentatives appear in the 1961 Book of ASTM Standards, Part 5.

[2] The revised tentatives appear in the 1961 Book of ASTM Standards, Part 5.

[3] 1958 Book of ASTM Standards, Part 5.

laundry type) .... 2.0 per cent
Water .... 45.0 per cent
Preservative.—Benzoic acid, 1.25 g per 100 g batch.

The starch is available at all grocery stores. It must be prepared by cooking with heat. For the cooking procedure, follow the instructions on the container. To the cooked starch, add the alumina and benzoic acid. The percentage of water prescribed should produce a primer mixture of thick paint consistency. It is normal to lose about 15 to 20 ml of water per 1000-g batch in the cooking operation. Water lost in the cooking procedure should be replaced for proper consistency control.

NOTE 2.—Application:

Apply the wet primer with a paint brush on the dry brick using about 2 oz per brick. Dry at normal room temperature and finally apply the refractory kaolin.

Definitions of Terms Relating to Refractories (C 71 – 55):[3]

New Definitions.—Under the heading "Materials," add the following new definitions:

Anthracite Coal Base Carbon Refractory.—A manufactured refractory comprised substantially of calcined anthracite coal.

Petroleum Coke Base Carbon Refractory.—A manufactured refractory comprised substantially of calcined petroleum coke.

Graphite Base Carbon Refractory.—A manufactured refractory comprised substantially of graphite.

Metallurgical Coke Base Carbon Refractory.—A manufactured refractory comprised substantially of metallurgical coke.

Insulating Fire Brick.—A refractory brick characterized by low thermal conductivity and low heat capacity.

Ladle Brick.—Fireclay brick suitable for lining ladles used to contain molten steel.

Molten Cast Refractory.—A solidified material made by melting refractory ingredients and pouring into molds.

Plastic Refractory.—A refractory material tempered with water which can be extruded and which has suitable workability to be pounded into place to form a monolithic structure.

Pouring Pit Refractory.—A refractory associated with the transfer or flow control of molten steel between furnace and mold.

Ramming Mix.—A refractory material tempered with water, which cannot be extruded but which has suitable properties to permit ramming into place to form a monolithic structure.

Neutral Refractories.—Refractories which are resistant to chemical attack by both acid and basic slags, refractories or fluxes at high temperatures.

Porosity.—The percentage of the total volume of a material occupied by both open and closed pores.

Method of Panel Spalling Test for High Duty Fireclay Brick (C 107 – 52):[3]

Section 4(a).—Change to read as follows:

4. (*a*) The test brick shall be primed with the alumina coating and laid up with refractory kaolin as outlined in Method C 38, Section 4.

Method of Panel Spalling Test for Super Duty Fireclay Brick (C 122 – 52):[3]

Section 4(a).—Change to read as follows:

4. (*a*) The test brick shall be primed with the alumina coating and laid up with refractory kaolin as outlined in Method C 38, Section 4.

Classification of Insulating Fire Brick (C 155 – 57):[3]

Table I.—Add a new Group 30, with reheat change test conducted at 2950 F (1620 C), with bulk density not greater than 68 lb per cu ft.

Section 3(a).—Change to read as follows:

3. (*a*) *Bulk Density (Weight per cu ft).*—Methods of Test for Size and Bulk Density of Insulating Fire Brick (C 437 – 59 T).

Method of Panel Spalling Test for Fireclay Plastic Refractories (C 180 – 52):[3]

Section 5(a).—Change to read as follows:

5. (*a*) The test brick shall be primed with the alumina coating and laid up with refractory kaolin as outlined in Method C 38, Section 4.

REVISION OF TENTATIVE REVISIONS

The committee recommends revisions as indicated of the following tentative revisions and their continuation as tentative:

Definition of Terms Relating to Refractories (C 71 - 55):[3]

Revised Definitions.—Revise the definitions in the Tentative Revisions[4] of June 1960, as follows:

Acid Refractories.—Refractories containing a substantial amount of silica which may react chemically with basic refractories, basic slags or basic fluxes at high temperatures.

Basic Refractories.[a]—Refractories whose major constituent is magnesia, lime or both and which react chemically with acid refractories, acid slags or acid fluxes at high temperatures.

[a] Commercial use of this term also includes refractories made of chrome ore or combinations of chrome ore and dead-burned magnesite.

Dead-burned (*adj.*).—The state of a basic refractory material resulting from a heat treatment which yields a product resistant to atmospheric hydration or carbonation.

Grain Magnesite.—Dead-burned magnesite in granular form of size suitable for refractory purposes.

Grog.—A granular material produced from calcined or burned refractories, usually alumino-silica.

Ground Fireclay Mortar.—A refractory mortar consisting of finely ground raw fire clay.

Burn (*n*).—The heat treatment to which refractory materials are subjected in the firing process.

Calcine (*n*).—Refractory material, often fire clay, which has been heated to eliminate volatile constituents and to produce desired physical changes.

Bloating of Refractories.—Substantial swelling produced by a heat treatment which causes the formation of a vesicular structure.

Specification for Refractories for Incinerators (C 106 - 51):[3]

Table I.—In the Tentative Revision of June 1960, change the requirement for the CaO, max, chemical composition of bonded silicon carbide from 0.5 to 0.6 per cent.

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends revisions as appended hereto[5] of the **Standard Specification for Fireclay-Base Castable Refractories for Boiler Furnaces and Incinerators (C 213 - 58).**[3]

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Methods of Test for:

Reheat Change of Carbon Brick and Shapes (**C 436 - 59 T**),

Size and Bulk Density of Insulating Fire Brick (**C 437 - 59 T**),

Thermal Conductivity of Plastic Refractories (**C 438 - 59 T**), and

Resistance to Thermal Spalling of Silica Brick (**C 439 - 59 T**).

Specifications for:

Insulating Fire Brick for Linings of Industrial Furnaces Operated with a Neutral or Oxidizing Atmosphere (**C 434 - 59 T**).

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee recommends that the tentative revisions[6] of Table I of the Standard Method of Test for Reheat Change of Refractory Brick (**C 113 - 46**)[3] be approved for reference to letter ballot of the Society for adoption as standard.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

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[4] 1960 Supplement to Book of ASTM Standards, Part 5.

[5] The revised tentative appears in the 1961 Book of ASTM Standards, Part 5.

[6] 1959 Supplement to Book of ASTM Standards, Part 5.

Specification for Refractories for Malleable Iron Furnaces with Removable Bungs and for Annealing Ovens (C 63 - 51):[3]

These specifications were extensively revised and are appended hereto in their proposed form:[7]

Specification for Refractories for Heavy Duty Stationary Boiler Service (C 64 - 51):[3]

These specifications were extensively revised and are appended hereto in their proposed form:[7]

Specification for Refractories for Moderate Duty Stationary Boiler Service (C 153 - 51):[3]

These specifications were extensively revised and are appended hereto in their proposed form:[7]

Method of Test for Reheat Change of Insulating Fire Brick (C 210 - 46):[3]

Table I.—Add the following new heating schedule for group 30 Insulating Fire Brick:

Elapsed Time from Start of Heating	Allowable Temperature Deviation	Group 30 2950 F Test
1 hr..........	±50 F	1750 F
1½ hr........	±35 F	2130 F
2 hr..........	±20 F	2370 F
2½ hr........	±15 F	2560 F
3 hr..........	±15 F	2680 F
3½ hr........	±15 F	2810 F
4 hr..........	±15 F	2880 F
4½ hr........	±15 F	2930 F
5 hr..........	±15 F	2950 F maintained for 24 hr

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards that have stood for six or more years without revision:

Methods of Test for:

Apparent Porosity, Water Absorption, Apparent Specific Gravity, and Bulk Density of Burned Refractory Brick (**C 20 - 46**),
Sieve Analysis and Water Content of Refractory Materials (**C 92 - 46**),
Crushing Strength and Modulus of Rupture of Insulating Fire Brick at Room Temperature (**C 93 - 54**),
Cold Crushing Strength and Modulus of Rupture of Refractory Brick and Shapes (**C 133 - 55**),
Size and Bulk Density of Refractory Brick (**C 134 - 41**),
Warpage of Refractory Brick and Tile (**C 154 - 41**),
Combined Drying and Firing Shrinkage of Fireclay Plastic Refractories (**C 179 - 46**),
Panel Spalling Test for Fireclay Plastic Refractories (**C 180 - 52**),
Workability Index of Fireclay Plastic Refractories (**C 181 - 47**),
Cold Bonding Strength of Air-Setting Refractory Mortar (Wet Type) (**C 198 - 47**), and
Refractoriness of Air-Setting Refractory Mortar (Wet Type) (**C 199 - 47**).

Classification of:

Single- and Double-Screened Ground Refractory Materials (**C 316 - 55**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[8]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Industrial Survey (A. Maupin, chairman) has completed the survey of refractories for the copper industry and is working on a revision of refractories for incinerators. Revision of surveys on refractories for blast furnaces, electric furnaces, portland cement industry and lime industry is planned.

Subcommittee II on Research (A. Allen, chairman) has proposed that the activities of the committee include a technical symposium as a part of Committee C-8 meetings and that a more active role be taken in visiting ceramic schools. The prime function of the committee would be to stimulate interest in research and to improve communications and education along these lines.

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[7] The revised standards appear in the 1961 Book of ASTM Standards, Part 5.

[8] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee III on Tests (S. M. Phelps, chairman) has investigated the possibility of simplifying reheat test schedules but concludes that no change is desired and no real problem exists.

Section A on Load (D. Stock, chairman) is investigating test procedures and equipment to determine hot modulus of rupture on refractories. Work is also being done on modulus of rupture of arch, wedge, and circle brick.

Ad Hoc Section on Long Time Load Testing (K. Babb, chairman) is investigating methods to determine creep in refractory materials under long time load tests. Semi-silica, 60 per cent alumina, and mullite brick are included in the first set of samples for comparative testing.

Section B on Spalling (L. Trostel, chairman) has made revisions in the four spalling test methods C 38, C 107, C 122, and C 180 for the use of a primer coat of alumina on test samples before kaolin mortar is applied. The precision of the spalling test is under study as well as a procedure for testing basic brick.

Section C on Temperature (J. L. Carruthers, chairman) reported on the new National Bureau of Standards pyrometric cone equivalent furnace. Results to date have not been encouraging.

Section E on Chemical Analysis (E. P. Pearson, chairman) has been investigating test methods for the determination of titania and sulfur in dolomitic refractories, boron in basic refractories, and sodium silicate in mortars and castables.

Task Group on Hydration (G. Eusner, chairman) is continuing work on periclase and magnesia products and is starting on testing of dolomitic materials.

Section F on Refractory Insulation (T. Brady, chairman) has proposed the addition of a group 30 classification to the Insulating Firebrick Classification C 155. Test requirements and procedures have been established.

Section G on Porosity and Permanent Volume Change (D. Rice, chairman) has been working on methods to determine bulk density, true specific gravity, and porosity of hydratable materials. Methods for determining end trueness of sleeves are being studied.

Section H on Mortars, Plastics, and Castables (G. H. Anthony, chairman) has proposed revisions of Methods C 268 and C 269 on the preparation of castable test samples to include the use of the consistency meter developed by the Mellon Institute.

Section K on Corrosion Test for Glass Tank Refractories (H. D. Prior, chairman) is investigating test methods to evaluate resistance of various refractories to glass corrosion. An interlaboratory series of tests is being prepared to determine the factors affecting test results.

Subcommittee IV on Heat Transfer (C. L. Norton, chairman) is reviewing information available on the use of a hot-wire method of measuring thermal conductivity and the measurement of conductivity on highly conductive materials.

Subcommittee VII on Specifications (J. D. Sullivan, chairman) has proposed tentative revisions of Methods C 63, C 64, C 153, and C 213.

Ad Hoc Section on Pouring Pit Refractories has withdrawn Specification C 435 for pouring pit refractories and resubmitted it as a classification.

Subcommittee VIII, Editorial (F. W. Schroeder, chairman) has reviewed the proposed revisions of the committee recommendations. Considerable revision of Method C 18 is in process.

Subcommittee IX on Classifications (J. A. Kayser, chairman) has under review proposed classifications or revision of classifications on basic refractories, steel pouring pit refractories, fireclay and high-alumina refractories and insulating firebrick.

Subcommittee XI on Special Refractories (A. Maupin, chairman) is continuing work on a classification for mullite and silicon carbide refractories.

Subcommittee XII on Carbon Refractories (E. B. Snyder, chairman) is further investigating permeability of refractories and a test for resistance of carbon refractories to alkali attack.

Subcommittee XV on Basic Granular Refractories (B. L. Dorsey, chairman) is working on a classification of magnesia and various tests to determine the physical and chemical properties of basic granular materials.

This report has been submitted to a letter ballot of the committee, which consists of 48 members; 43 members returned their ballots, of whom 42 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. J. HAZEL,
Chairman.

W. O. BRANDT,
Secretary.

REPORT OF COMMITTEE C-9
ON
CONCRETE AND CONCRETE AGGREGATES*

Committee C-9 on Concrete and Concrete Aggregates held two meetings during the year: on June 29, 1960, during the Annual Meeting of the Society in Atlantic City, N. J., and on Dec. 9, 1960, in Denver, Colo.

At its June meeting, the committee adopted memorials to H. F. Thomson, a member of the committee since 1952, who died on November 7, 1959; P. D. Miesenhelder, a member of the committee since 1951, who died on November 17, 1959; P. J. Freeman, an Honorary Member and member of the committee since 1923, who died in November, 1959; and James Murray, a member of the committee since 1952, who died on March 5, 1960. At its December meeting, the committee adopted a memorial to H. F. Clemmer, a member of the committee since 1923, who died on October 5, 1960.

At its December meeting, the Executive Subcommittee elected Fred Hubbard to Honorary Membership.

The Sanford E. Thompson Award, established by the committee in 1938, will be made this year to Messrs. Leonard Pepper and Bryant Mather, of the U. S. Army Engineer Waterways Experiment Station, Concrete Division, for their paper entitled "Effectiveness of Mineral Admixtures in Preventing Excessive Expansion of Concrete Due to Alkali-Aggregate Reaction," which was presented at the 62nd Annual Meeting of the Society, June, 1959, and was published in the 1959 *Proceedings.*

NEW TENTATIVES

The committee recommends for publication as tentative the following method and specification as appended hereto[1]:

Tentative Method of Test for:

Static Young's Modulus of Elasticity and Poisson's Ratio in Compression of Cylindrical Concrete Specimens.

Tentative Specifications for:

Single-Use Molds for Forming 6 by 12-in. Concrete Compression Test Cylinders.

REVISION OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Method of Test for Rate of Hardening of Mortars Sieved from Concrete Mixtures by Proctor Penetration Resistance Needles (C 403 – 57 T):[2]

Revise as appended hereto.[3]

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] The new tentatives appear in the 1961 Book of ASTM Standards, Part 4.

[2] 1958 Book of ASTM Standards, Part 4.

[3] The revised tentative appears in the 1961 Book of ASTM Standards, Part 4.

Tentative Method of Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88 – 59 T):[4]

Section 5(c).—Delete this paragraph.

Section 6(b).—Change to read as follows:

> (*b*) *Drying Samples After Immersion.*—After the immersion period the aggregate sample shall be removed from the solution, permitted to drain for 15 ± 5 min and placed in the drying oven. The temperature of the oven shall have been brought previously to 221 to 230 F (105 to 110 C). The samples shall be dried at the specified temperature until constant weight has been achieved. During the drying period the samples shall be removed from the oven, cooled to room temperature, and weighed at time intervals of not less than 4 hr nor more than 18 hr. Constant weight may be considered to have been achieved when two successive weights for any one sample, made as described above, differ by less than 0.1 g, in the case of fine aggregate samples; or by less than 1.0 g in the case of coarse aggregate samples. After constant weight has been achieved the samples shall be allowed to cool to room temperature, when they shall again be immersed in the prepared solution as described in Paragraph (*a*).

Section 7.—In Paragraph (*b*) change the first sentence to read as follows:

> After the sodium sulfate or magnesium sulfate has been removed, each fraction of the sample shall be dried to constant weight at 221 to 230 F (105 to 110 C), and weighed. The fine aggregate shall be sieved over the same sieve on which it was retained before the test, and the coarse aggregate shall be sieved over the sieve shown below for the appropriate size of particle.

Delete Paragraph (*c*), including the note.

Section 9.—In Paragraph (*b*), delete the initial phrase "Except in the case of ledge rock."

Delete Paragraph (*e*), and reletter Paragraph (*f*) as Paragraph (*e*).

Tentative Method of Test for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar Bar Method) (C 227 – 60 T):[5]

Section 9.—Change Item (5) to read: "(5) Any relevant information concerning the preparation of aggregates including the grading of the aggregate differing from that given in Section 4(*b*)."

Add a new Item (8) to read: "(8) Type, source, proportions, and chemical analyses, including Na_2O and K_2O, of any pozzolans employed in the tests."

Tentative Method of Test for Potential Reactivity of Aggregates (Chemical Method) (C 289 – 57 T):[2]

Section 1.—In line 1, insert the word "chemical" before the last word.

Section 4(a).—Add the following sentence at the end of this paragraph: "Store the solution in a polyethylene container and protect from contamination by carbon dioxide."

Section 6(c).—Insert an additional sentence immediately prior to the last sentence to read as follows:

> If inspection of the sample indicates the presence of silty or clayey coatings on particles, repeat the washing and drying procedure, and sieve as before over the No. 100 sieve.

Section 7(a).—In the second and third sentences, insert the words "by means of a pipette and buret" before the word "add."

Section 7(e).—At the end of this section, add a new note reading as follows:

> NOTE.—If the diluted filtrate is not to be analyzed within 4 hr following completion of the filtration, transfer the solution to a clean, dry polyethylene container and close the container by means of a stopper or tight-fitting cap or lid.

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[4] 1959 Supplement to Book of ASTM Standards, Part 4.

[5] 1960 Supplement to Book of ASTM Standards, Part 4.

Section 11.—Insert a sentence at the end of Paragraph (*a*) to read as follows:

> Do not use a standard silica solution older than one year, since dissolved ionic silica in such a solution slowly polymerizes, causing spuriously low photometric readings.

In Paragraph (*b*), replace the present second sentence with the following: "If the solution is not clear, filter through a tight filter paper. Store the solution in a chemically resistant glass bottle coated on its interior with paraffin, or in a plastic bottle."

Section 12(*a*).—In the last sentence, change "5 min" to read "5.0 ± 0.1 min."

Section 15.—In the first sentence of Paragraph (*a*), change "(**1–5**)" to read "(**1–7**)."

Insert a new Paragraph (*c*) to read as follows, relettering the present Paragraph (*c*) as Paragraph (*d*):

> (*c*) Data show that aggregates represented by data points falling to the right of the boundary line shown in Fig. 2 and above a line extending from about point $Rc = 75$, $Sc = 70$ to a point $Rc = 125$, $Sc = 1000$ may be sufficiently alkali-reactive to produce comparatively low expansion when tested in accordance with Method of Test for Potential Alkali Reactivity of Cement-Aggregate Combinations (ASTM Designation: C 227). In this circumstance, the results of the chemical test should be accepted as indicating a potentially deleterious degree of alkali reactivity unless the innocuous character of the aggregate is demonstrated by supplementary tests in accordance with Method C 227 of several mixtures of the test aggregate and an essentially inert aggregate, such as properly graded quartz, Ottawa sand, or chert-free limestone. The mixtures should represent a series of proportions of the test aggregate and essentially inert aggregate ranging from about 5:95 to 50:50 by weight.

References.—Add references (**6**) and (**7**) as follows:

(**6**) R. C. Mielenz and E. J. Benton, "Evaluation of the Quick Chemical Test for Alkali Reactivity of Concrete Aggregate," ***Bulletin 171***, Highway Research Board, p. 1 (1958).

(**7**) Bernard Chaiken and W. J. Halstead, "Correlation Between Chemical and Mortar Bar Tests for Potential Alkali Reactivity of Concrete Aggregates," *Public Roads*, Vol. 30, p. 177 (1959).

Tentative Method of Test for Resistance of Concrete Specimens to Rapid Freezing and Thawing in Water (C 290 – 57 T):[2]

Section 2(*a*).—In Item (*1*) insert the phrase, "continuously and automatically," after the word "produce" in line 8 and add the following new sentence to the end of the paragraph:

> In the event that the equipment does not operate automatically, provision shall be made for either its continuous manual operation on a 24-hr a day basis or for the storage of all specimens in a frozen condition when the equipment is not in operation.

Section 3.—Change Section 3 including the note to read as follows:

> 3. Conformity with the requirements for the freezing-and-thawing cycle shall be based on temperature measurements of control specimens of similar concrete to the specimens under test in which suitable temperature-measuring devices have been imbedded. The position of these control specimens shall be changed frequently in such a way as to indicate the extremes of temperature variation at different locations in the specimen cabinet. The nominal freezing-and-thawing cycle for this method shall consist of alternately lowering the temperature of the specimens from 40 to 0 F and raising it from 0 to 40 F in not less than 2 nor more than 4 hr, of which not less than 25 per cent of the time is used for thawing (Note). At the end of the cooling period the temperature at the centers of the specimens shall be 0 ± 3 F, and at the end of the heating period shall be 40 ± 3 F, with no specimen at any time reaching a temperature lower than −3 F nor higher than 43 F. The time required for the temperature at the center of any single specimen to be reduced from 37 to 3 F shall be not less than half the length of the cooling period, and the time required for the temperature at the center of any single specimen to be raised from 3 to 37 F shall be not less than half the length of the heating period. For specimens to be compared with each other, the time required to change the temperature at the center of any specimen from

35 to 10 F shall not differ by more than one-sixth the length of the cooling period from the time required for any other specimen, and the time required to change the temperature at the center of any specimen from 10 to 35 F shall not differ by more than one-third the length of the heating period from the time required for any other specimen. The difference between the temperature at the center of a specimen and the temperture at its surface shall at no time exceed 50 F.

NOTE.—In most cases, uniform temperature and time conditions can be controlled most conveniently by maintaining a capacity load of specimens in the equipment at all times. In the event that a capacity load of test specimens is not available, dummy specimens can be used to fill empty spaces. This procedure also assists greatly in maintaining uniform fluid level conditions in the specimen and solution tanks. The dummy specimens should be replaced from time to time as they deteriorate under exposure.

The testing of concrete specimens composed of widely varying materials or with widely varying thermal properties, in the same equipment at the same time, may not permit adherence to the time-temperature requirements for all specimens. It is suggested, when practical, that such specimens be tested at different times and that appropriate adjustments be made to the equipment.

Section 5.—Add a new Paragraph (*d*) to read as follows:

(*d*) If, due to equipment breakdown or for other reasons, it becomes necessary to interrupt the cycles for a protracted period, the specimens shall be maintained in a frozen condition in the containers and surrounded by ice, if possible. If it is not possible to store the specimens in their containers, they shall be wrapped and sealed, in as wet a condition as possible, in moistureproof material to prevent dehydration and stored in a refrigerator or cold room maintained at 0 ± 3 F. Judgment must be exercised in determining what is a protracted period. In general specimens remaining in a thawed condition for more than two cycles is undesirable but a longer period may be permissible if this occurs only once or twice during a test.

Section 6(*a*).—Change to read as follows:

6. (*a*) *Relative Dynamic Modulus of Elasticity, per cent.*—Numerical values of relative dynamic modulus of elasticity shall be calculated by the following equation:

$$P_c = \frac{n_1^2}{n^2} \times 100$$

where:

P_c = relative dynamic modulus of elasticity, per cent, after c cycles of freezing and thawing,

n = fundamental transverse frequency at 0 cycles of freezing and thawing, and

n_1 = fundamental transverse frequency after c cycles of freezing and thawing.

NOTE.—This calculation of relative dynamic modulus of elasticity is based on the assumption that the weight and dimensions of the specimen remain constant throughout the test. This assumption is not true in many cases due to disintegration of the specimen. However, if the test is to be used to make comparisons between the relative dynamic moduli of different specimens or of different concrete formulations, P_c as defined is adequate for the purpose.

Tentative Method of Test for Resistance of Concrete Specimens to Rapid Freezing in Air and Thawing in Water (C 291 – 57 T):[2]

Section 2(*a*).—Revise as indicated above for Section 2(*a*) of Method C 290.

Section 3.—Change Paragraph (*a*) to read as follows:

3. (*a*) Conformity with requirements of Paragraphs (*b*) and (*c*) shall be based on temperature measurements of control specimens of similar concrete to the specimens under test in which suitable temperature measuring devices have been imbedded. The position of these control specimens shall be changed frequently in such a way as to indicate the extremes of temperature variation at different locations in the specimen cabinet.

NOTE.—In most cases, uniform temperature and time conditions can be controlled most conveniently by maintaining a capacity load of specimens in the equipment at all times. In the event that a capacity load of test specimens is not available, dummy specimens can be used to fill empty space. The dummy specimens shall be replaced from time to time as they deteriorate under exposure.

The testing of concrete specimens composed of widely varying materials or with widely varying thermal properties in the same equipment at the same time, may not permit adherence to the time-temperature requirements for all specimens. It is suggested, when practical, that such specimens be tested at different times and that appropriate adjustments be made to the equipment.

Change Paragraph (*b*) to read as follows:

(*b*) The nominal freezing-and-thawing cycle for this method shall consist of alternately lowering the temperature of the specimens from 40 to 0 F in a period of not more than 3 hr and raising it from 0 to 40 F in a period of not more than 1 hr. At the end of the cooling period the temperature at the centers of the specimens shall be 0 ± 3 F, and at the end of the heating period shall be 40 ± 3 F, with no specimen at any time reaching a temperature lower than −3 F nor higher than 43 F. The time required for the temperature at the center of any single specimen to be reduced from 37 to 3 F shall be not less than half the length of the cooling period, and the time required for the temperature of any single specimen to be raised from 3 to 37 F shall be not less than half the length of the heating period. For specimens to be compared with each other, the time required to change the temperature at the center of any specimen from 35 to 10 F and from 10 to 35 F shall not differ by more than one-sixth the length of the cooling and heating periods respectively, from the time required for any other specimen.

Delete Paragraph (*e*).

Section 5.—Add a new Paragraph (*d*) to read as follows:

(*d*) If, due to equipment breakdown or for other reasons, it becomes necessary to interrupt the cycles for a protracted period, the specimens shall be wrapped and sealed in moistureproof material to prevent dehydration, and maintained in a frozen condition in a refrigerator or cold room maintained at 0 ± 3 F. In general, specimens remaining in a thawed condition for more than two cycles is undesirable but a longer period may be permissible if this occurs only once or twice during a test.

Section 6 (*a*).—Revise to read as indicated above for Section 6(*a*) of Method C 290.

Tentative Method of Test for Resistance of Concrete Specimens to Slow Freezing and Thawing in Water or Brine (C 292 – 57 T):[2]

Section 3.—Change to read as follows:

3. Conformity with the requirements for the freezing-and-thawing cycle shall be based on temperature measurements of control specimens of similar concrete to the specimens under test in which suitable temperature-measuring devices have been imbedded. The position of these control specimens shall be changed frequently in such a way as to indicate the extremes of temperature variation at different locations in the specimen cabinet. The nominal freezing and thawing cycle for this method shall consist of alternately lowering the temperature of the specimens from 73.4 to 0 F in 18 to 24 hr and raising the temperature of the specimens from 0 to 73.4 F in 18 to 24 hr (Note). At the end of the cooling period the temperature at the centers of the specimens shall be 0 ± 3 F, and at the end of the heating period, the temperature at the centers of the specimens shall be 73.4 ± 3 F, with no specimen at any time reaching a temperature lower than −3 F nor higher than 76.4 F. The time required for the temperature at the center of any single specimen to be reduced from 70.4 to 3 F or raised from 3 to 70.4 F shall be not less than 18 nor more than 24 hr. For specimens to be compared with each other, the time required to change the temperature at the center of any single specimen from 60 to 10 F and from 10 to 60 F shall not differ by more than 30 minutes from that required for any other specimen. The difference between the temperature at the center of a specimen and the temperature at its surface shall at no time exceed 50 F.

NOTE.—In most cases, uniform temperature and time conditions can be controlled most conveniently by maintaining a capacity load of specimens in the equipment at all times. In the event that a capacity load of test specimens is not available, dummy specimens can be used to fill empty spaces. This procedure also assists greatly in maintaining uniform fluid level conditions in the specimen and solution tanks. The dummy specimens shall be replaced from time to time as they deteriorate under exposure.

The testing of concrete specimens composed of widely varying materials or with widely varying thermal properties, in the same equipment at the same time, may not permit adherence to the time-temperature requirements for all specimens. It is suggested, when practical, that such specimens be tested at different times and that appropriate adjustments be made to the equipment.

Section 5.—Add the following two sentences to the end of Paragraph (*c*):

This provision applies whenever circumstances arise that would require any freezing-and-thawing cycle to take more than 48 hr, such as over weekends. At such times the freezing period shall be extended to include all the extra time.

Add a new Paragraph (*d*) as indicated above for Section 5(*d*) of Method C 290.

Section 6(a).—Revise to read as indicated above for Section 6(*a*) of Method C 290.

Tentative Method of Test for Resistance of Concrete Specimens to Slow Freezing in Air and Thawing in Water (C 310 – 57 T):[2]

Section 3.—Change Section 3 to read as follows:

3. (*a*) Conformity with the requirements of Paragraph (*b*) shall be based on temperature measurements of control specimens of similar concrete to the specimens under test in which suitable temperature-measuring devices have been imbedded. The position of these control specimens shall be changed frequently in such a way as to indicate the extremes of temperature variation at different locations in the specimen cabinet.

NOTE 1.—In most cases, uniform temperature and time conditions can be controlled most conveniently by maintaining a capacity load of specimens in the equipment at all times. In the event that a capacity load of test specimens is not available, dummy specimens can be used to fill empty space. The dummy specimens shall be replaced from time to time as they deteriorate under exposure.

The testing of concrete specimens composed of widely varying materials or with widely varying thermal properties, in the same equipment at the same time, may not permit adherence to the time-temperature requirements for all specimens. It is suggested when practical that such specimens be tested at different times and that appropriate adjustments be made to the equipment.

(*b*) The nominal freezing-and-thawing cycle for this method shall consist of alternately lowering the temperature of the specimens from 40 to 0 F and raising it from 0 to 40 F, with the cooling period taking a total time of 18 ± 2 hr and the heating period taking a total time of 6 ± 1 hr. During the cooling period the temperature at the centers of the specimens shall be reduced from 37 to 3 F in a period of not less than 5 nor more than 7 hr and shall remain at 0 ± 3 F for the remainder of the cooling period. During the heating period, the temperature at the centers of all specimens shall be raised from 3 to 37 F in not less than 1½ hr nor more than 2 hr and shall remain at 40 ± 3 F for the remainder of the heating period. For specimens to be compared with each other, the time required to change the temperature at the center of any specimen from 35 to 10 F and from 10 to 35 F shall not differ by more than 30 min from the time required for any other specimen (Note 2). The temperature of the thawing water, and the difference between the temperature at the center of a specimen and the temperature at its surface shall at no time exceed 50 F.

NOTE 2.—To meet the temperature requirements some equipment may require two thermostats operated alternately by electric timers, to control the temperature of the cooling medium: one set at somewhat below 0 F for the initial cooling period and one set at 0 F to maintain this temperature for the remainder of the 18 hr.

(*c*) The cooling may be accomplished in either of two ways: (*1*) forced circulation of cold air around the specimens, or (*2*) immersion of the specimens in watertight containers in circulating brine or other cold liquid. In the first method, the rate of air circulation should be the minimum required to ensure equal exposure of all specimens, so that transfer of moisture from the specimens to the refrigerating areas will be held to a minimum. Likewise refrigerating areas in the cooling chamber should be as large as practicable to minimize moisture transfer. In the second method, each specimen shall be placed in a container in a manner that will prevent contact between the specimen and the container and with the bottom of the specimen supported on wood blocks. The container shall be partially immersed in the circulating liquid in such a manner that the level of the top of the specimen is below the level of the circulating liquid.

(*d*) The period of transition from the cooling period to the heating period of the cycle or vice versa shall not exceed 10 min except when specimens are being tested in accordance with Section 5(*b*). The surfaces shall not be allowed to become dry during the transition from the heating to the cooling period of the cycle.

Section 5.—Add the following two sentences to the end of Paragraph (*c*):

This provision applies whenever circumstances arise that would require any freezing-and-thawing cycle to take more than 24 hr such as over weekends. At such times the freezing period shall be extended to include all of the extra time.

Add a new Paragraph (*d*) to read as indicated above for Section 5(*d*) of Method C 291.

Section 6(*a*).—Revise to read as indicated above for Section 6(*a*) of Method C 290.

Tentative Method of Test for Potential Volume Change of Cement-Aggregate Combinations (C 342 - 55 T):[2]

Section 2(*c*).—Insert after the first sentence: "They shall be so constructed that, when using for storing specimens, the loss of moisture is prevented by a tight-fitting cover or by sealing, or by both."

Change the last sentence of Paragraph (*c*) to read as follows:

Provision shall be made for supporting the specimens ½ in. above the bottom of the container in such manner that the weight of the specimens is not supported by the gage studs, and the specimens are kept vertical, spaced uniformly, and free from contact with the walls of the container.

Change the note at the end of Paragraph (*c*) to read as follows:

NOTE.—Removable corrosion-resistant 20- to 30-gage sheet-metal spacers have been found to be satisfactory to space and support the specimens. The top spacer is a rectangular strip 1⅛ in. in width and 3⅜ in. in length to fit into the interior cross-section dimension of the container. The strip is perforated along the longitudinal center line with three holes of size to permit insertion of the gage studs at ends of specimens. The hole for the center specimen is placed at the center of the strip, and the centers of the other holes are placed 1⅛ in. to either side of the center. The bottom spacer is the same except for an added length of ½ in. at each end that are turned at right angles to the face of the strip to form legs to support spacers ½ in. above bottom of container.

Section 3(*a*).—Change to read as follows:

(*a*) The temperature of the dry materials, the molding room, and the measuring room shall be between 68 and 81.5 F (20 to 27.5 C). The temperature of the mixing water and the moist closet or moist room shall be maintained at 73.4 ± 3 F (23 ± 1.7 C). The relative humidity of the molding room shall be not less than 50 per cent.

Section 4.—Change the heading to read "Selection and Preparation of Materials," and replace the present Section 4 with the following:

4. (*a*) *Selection of Aggregate.*—Materials proposed for use as fine aggregate in concrete shall be processed as described in Paragraph (*b*) with a minimum of crushing. Materials proposed for use as coarse aggregate in concrete shall be processed by crushing to produce as nearly as practical a graded product from which a sample can be obtained. The sample shall have the grading as prescribed in Paragraph (*b*) and be representative of the composition of the coarse aggregate as proposed for use (Notes 1 and 2).

NOTE 1.—When a given quarried material is proposed for use, both as coarse and as fine aggregate, it will be tested only by selection of an appropriate sample crushed to the fine aggregate sizes, unless there is reason to expect that the coarser size fractions have a different composition than the finer sizes and that these differences might significantly affect expansion due to reaction with the alkalies in cement. In this case, the coarser size fractions should be tested in a manner similar to that employed in testing the fine aggregate sizes.

NOTE 2.—Coarse aggregate crushed to sand size may give accentuated expansion, owing to the increased surface exposed upon crushing. Therefore, if coarse aggregate tested by this method produces an excessive amount of expansion, the material shall not be classed as objectionably reactive with alkali unless tests of concrete specimens confirm the findings of the tests of the mortar.

(*b*) *Preparation of Aggregate.*—Fine aggregate shall be tested in a grading meeting the requirements of the specifications for the project. Fine aggregates being tested for reasons other than to determine compliance with individual specifications, and all coarse aggregates to which this method of test is applied shall be graded in accordance with the requirements prescribed in Table I. Aggregates in which sufficient quantities of the sizes specified in Table I do not exist shall be crushed until the required material has been produced. In the case of aggregates containing insufficient amounts of one or more of the larger sizes listed in Table I, and if no larger material is available for crushing, the first size in which sufficient material is available shall contain the cumulative percentage of material down to that size as determined from the grading specified in Table I. When such procedures are required, special note shall be made thereof in the test report. After the aggregate has been separated into the various sieve sizes, each size shall be washed with a water spray over the sieve to remove adhering dust and fine particles from the aggregate. The sieve splits shall then be dried and recombined in the quantity required for one batch of mortar to the grading prescribed in Table I.

TABLE I.—GRADING REQUIREMENTS.

Sieve Size		Percentage by Weight
Passing	Retained on	
No. 4 (4760-μ)	No. 8 (2380-μ)	10
No. 8 (2380-μ)	No. 16 (1190-μ)	25
No. 16 (1190-μ)	No. 30 (590-μ)	25
No. 30 (590-μ)	No. 50 (297-μ)	25
No. 50 (297-μ)	No. 100 (149-μ)	15

(*c*) *Selection of Cement.*—Cement (Note 3) for use in this method shall meet the requirements of the specifications for the project and shall be from the source or sources which will or are expected to supply the cement to be used on the project. Cement for use in tests made for reasons other than to determine compliance of cement-aggregate combinations with individual specifications shall conform to the requirements of the Specifications for Portland Cement (ASTM Designation: C 150) (Note 4). Cement for use in this method of test shall be passed through a No. 20 (840 μ) sieve to remove lumps before use.

NOTE 3.—If several cements are to be used or may be used in the work, it is recommended that each of these cements be tested in combination with each of the proposed aggregates.

NOTE 4.—It is recommended that each aggregate subjected to this method of test be used with both high- and low-alkali portland cements; other combinations with pozzolans, for example, may be used if requested. A low-alkali portland cement is defined in the Specifications C 150 as one containing not more than 0.60 per cent alkalies. Cements of alkali contents as low and as high as can be obtained should be used.

Section 5.—Change Paragraph (*b*) to read as follows:

Thinly cover the molds with mineral oil; after this operation set the gage studs, taking care to keep them clean and free of oil. Apply a mixture of three parts of paraffin to five parts of rosin by weight, heated between 230 and 248 F (110 and 120 C) to the outside contact lines of the molds to make the molds watertight."

In Paragraph (*e*), line 6, change the word "plugs" to "studs."

Section 7.—Change the heading of Section 7 to read "Calculation."

Section 8.—Renumber the present Section 8 as Section 10. Add a new Section 8 headed "Examination at End of Test" to read as follows:

8. After the final length change measurements have been made, each specimen shall be tested for warping and shall be examined.

(*a*) *Warping.*—The warping, if any, that each specimen has manifested during the test period, shall be determined by placing the specimen on a plane surface and measuring the maximum separation between the specimen and the surface. The specimen shall be placed so that its ends curve down to the reference surface and the measurement made to the nearest 0.01 in.

(*b*) *Examination.*—Items to be noted in the examination include:

(*1*) Presence, location, and type of pattern of cracking,

(*2*) Appearance of surfaces, surface mottling, and

(*3*) Surficial deposits or exudations, their nature, thickness and continuity.

When it is believed that additional features of noteworthy importance are likely to be revealed thereby, a further examination of the interior of the specimen may be made, preferably by a petrographer. In such cases, the presence of gel-filled pores and fissures, evidence of reaction with aggregate particles of specific types and the nature of such evidence, and the properties of the reaction products are among the items to be noted.

Section 9.—Add a new Section 9 headed "Report" to read as follows:

9. The report shall include the following:

(*1*) Type and source of aggregate,

(*2*) Type and source of portland cement,

(*3*) Chemical analysis of the cement including alkali content as per cent potassium oxide (K_2O), sodium oxide (Na_2O), and calculated sodium oxide (Na_2O) equivalent,

(*4*) Grading of the aggregate if not that given in Section 4,

(*5*) Type, source, proportions, and chemical analysis, including K_2O and Na_2O, of any pozzolans employed in the tests,

(*6*) Average length change in per cent at each reading of the specimens,

(*7*) Any relevant information concerning preparation of the aggregates,

(*8*) Any significant features revealed by examination of the specimens during and after test, and

(*9*) Amount of mixing water, expressed as percentage by weight of cement.

Tentative Methods of Sampling and Testing Fly Ash for Use as an Admixture in Portland Cement Concrete (C 311 - 59 T):[4]

Section 27.—In Paragraphs (*a*), (*b*), and (*c*) change "4.5 ± 0.5 per cent of air" to read "5.5 ± 0.5 per cent of air."

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Method of Test for Compressive Strength of Molded Concrete Cylinders (C 39 - 59):[2]

Section 3.—Insert a new second sentence and note as follows: The ends of compression test specimens that are not plane within 0.002 in. shall be capped (Note).

NOTE.—For methods of capping concrete compression test specimens see Methods C 192 and C 31.

Standard Methods of Securing, Preparing, and Testing Specimens from Hardened Concrete for Compressive and Flexural Strengths (C 42 - 60):[4]

Section 7.—In the table in Paragraph (*f*), change the strength correction factor for $L/D = 1.00$ from "0.85" to "0.89."

Standard Specifications for Ready-Mixed Concrete (C 94 - 58):[2]

Section 9.—Replace the present Paragraph (*h*) with the following:

(*h*) Concrete delivered in cold weather shall have the applicable minimum temperature indicated in the following table. (The purchaser shall inform the producer as to the type of construction for which the concrete is intended.)

Air Temperature, deg Fahr	Minimum Concrete Temperature, deg Fahr	
	Thin Sections and Unformed Slabs	Heavy Formed Sections and Mass Concrete
30 to 45	60	50
0 to 30	65	55
Below 0	70	60

The maximum temperature of concrete produced with heated aggregates, heated water, or both, shall at no time during its production or transportation exceed 90 F (Note 5).

NOTE 5.—When cement is at a low temperature and hot mixing water is used, flash set may occur if hot water is brought in direct contact with the cement.

Add a new Paragraph (*i*) to read as follows:

(*i*) Every effort shall be made to maintain temperature of concrete produced during hot weather as low as possible. In some situations difficulty may be encountered with concrete at temperatures approaching 90 F, and every effort should be made to keep the concrete at a lower temperature.

REVISION OF STANDARDS AND REVERSION TO TENTATIVES

The committee recommends revisions as indicated of the following standards and their reversion to tentative:

Standard Specifications for Concrete Aggregates (C 33 – 59):[4]

Section 1.—Change the Scope to read: "These specifications cover fine and coarse aggregate other than lightweight aggregate for use in concrete.[3]"

Section 4.—In Paragraph (*b*) (*3*), insert the following sentence just before the present last sentence: "Care should be taken not to lose fine material."

Table I.—Add the following sub-items under Coal and lignite, and delete Note[b]:

Coal and lignite	
Where surface appearance of concrete is of importance	0.5
All other concrete	1.0

Table III.—Add the following sub-items under Coal and lignite, and delete the present Note[b]:

Coal and lignite	
Where surface appearance of concrete is of importance	0.5
All other concrete	1.0

Replace the line that now reads "Chert that will readily disintegrate (soundness test, five cycles)...1.0" to read as follows and add new Notes[b] and [c]:

Chert as an impurity[b] that will disintegrate in five cycles of the soundness test, or 50 cycles of freezing and thawing (0 F to 40 F)[c]; or that has a specific gravity, saturated-surface-dry, of less than 2.35:	
Severe exposure	1.0
Mild exposure	5.0

[b] These limitations apply only to aggregates in which chert appears as an impurity. They are not applicable to gravels that are predominantly chert. Limitations on soundness of such aggregates must be based on service records in the environment in which they are used.

[c] Disintegration is considered to be actual splitting or breaking as determined by visual examination.

Section 12(j).—Add the following sentences at the end of Paragraph (*j*): "Only material that is brownish-black, or black, shall be considered coal or lignite. Coke shall not be classed as coal or lignite."

Standard Method of Test for Amount of Material Finer Than No. 200 Sieve in Aggregate (C 117 – 49)[2] (Jointly with Committee D-4):

Revise as appended hereto.[6]

Standard Method of Test for Sieve Analysis of Fine and Coarse Aggregates (C 136 – 46)[2] (Jointly with Committee D-4): Revise as appended hereto.[6]

TENTATIVE REVISIONS OF STANDARDS

The committee recommends revisions as indicated of the following standards for publication as tentative:

Standard Methods of Securing, Preparing, and Testing Specimens from Hardened Concrete for Compressive and Flexural Strengths (C 42 – 60):[4]

Section 7(f).—Change to read as follows:

(*f*) *Calculations and Report.*—The direction of the application of the load with reference to direction of compaction of the concrete in the structure shall be reported. The compressive strength of each specimen shall be calculated to the nearest 10 psi based on the average diameter of the specimen. If the ratio of length to diameter of a specimen is appreciably less than two, allowance for the ratio of length to diameter shall be made by multiplying the compressive strength by the applicable correction factors given in the following table:

Ratio of Length of Cylinder to Diameter (l/d)	Strength Correction Factor Normal Weight Concrete[a]	Lightweight Concrete[b]
1.75	0.98	0.98
1.50	0.97	0.96
1.25	0.93	0.94
1.00	0.89	0.92

[a] These correction factors are not applicable to autoclaved concrete or to concrete dry at the time of loading.

[b] For concretes weighing between 100 and 120 lb per cu ft.

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[6] The revised methods appear in the 1961 Book of ASTM Standards, Part 4.

Values not given in the table shall be determined by interpolation. The correction factors are applicable for nominal concrete strengths from 2000 to 6000 psi.

Standard Specifications for Ready-Mixed Concrete (C 94 – 58):[2]

Section 9(*c*).—Change to read as follows:

(*c*) When a stationary mixer is used for the complete mixing of the concrete, mixing time shall be measured from the time all the solid materials are in the drum. The batch shall be so charged into the mixer that some water will enter in advance of the cement and aggregate, and all water shall be in the drum by the end of the first one fourth of the specified mixing time.

Where no mixer performance tests are made, the acceptable mixing time for mixers having capacities of 1 cu yd or less shall be not less than 1 min. For mixers of greater capacity, this minimum shall be increased 15 sec for each cubic yard or fraction thereof of additional capacity.

Where mixer performance tests have been made on given concrete mixtures in accordance with the testing program set forth in the following paragraphs, and the mixers have been charged to their rated capacity, the acceptable mixing time may be reduced for those particular circumstances to a point at which satisfactory mixing defined as follows shall have been accomplished:

(*1*) Samples of concrete for comparative purposes shall be obtained immediately after arbitrarily designated mixing times, in accordance with one of the following procedures:

Alternate Procedure 1.—The mixer may be stopped, and the required samples removed by any suitable means from the concrete at equal distances from the front and back of the drum, or

Alternate Procedure 2.—As the mixer is being completely emptied, appropriate samples may be obtained from the second and ninth-tenth portions of the batch. Any appropriate method may be used, provided the samples are representative of widely separated portions, but not the very ends of the batch.

(*2*) The samples of concrete shall be tested in accordance with methods listed in Section 18, and differences in test results for the two samples shall not exceed the following:

Weight per cu ft calculated to an air-free basis	1 lb
Air content	1.0 per cent by volume of the concrete
Slump:	
3 in. or less	¾ in.
more than 3 in	¼ of average for two samples
Coarse aggregate content for portion of each sample by weight retained on No. 4 sieve	6.0 per cent by weight of the respective samples, based on the weight of that portion of each sample retained on the No. 4 (4760-micron) sieve
Cement Content or Unit Weight of Mortar:	
Alternate 1.—Cement content, average for each sample determined by Willis-Hime procedure[1]	7 per cent
Alternate 2.—Unit weight of mortar, from calculated average weight per cu ft of air-free mortar determined by the Bureau of Reclamation procedure[2]	0.8 per cent
Compressive strength, average strength at 7 days of all cylinders tested, with 3 cylinders to be made and tested from each sample	7½ per cent

(*3*) Mixer performance tests shall be repeated whenever the appearance of the concrete or the coarse aggregate content of samples selected as outlined in this section indicates that adequate mixing has not been accomplished.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for:

Lightweight Aggregates for Insulating Concrete (C 332 – 56 T),[2] and

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[1] "Proposed Tentative Method of Test for Cement Content of Freshly Mixed Concrete," ASTM BULLETIN No. 239, July 1959, p. 48.

[2] "Test for Variability of Constituents in Concrete," Designation 26, Bureau of Reclamation *Concrete Manual*, 6th edition, p. 447.

Cotton Mats for Curing Concrete (**C 440 – 59 T**).[4]

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Method of Test for:

Measuring Mortar-Making Properties of Fine Aggregate (**C 87 – 58 T**),
Lightweight Pieces in Aggregate (**C 123 – 57 T**),
Clay Lumps in Natural Aggregates (**C 142 – 55 T**),
Water Retention Efficiency of Liquid Membrane-Forming Compounds and Impermeable Sheet Materials for Curing Concrete (**C 156 – 55 T**),
Comparing Concretes on the Basis of the Bond Developed with Reinforcing Steel (**C 234 – 57 T**),
Scratch Hardness of Coarse Aggregate Particles (**C 235 – 57 T**),
Ball Penetration in Fresh Portland Cement Concrete (**C 360 – 55 T**),
Abrasion Resistance of Concrete (**C 418 – 58 T**), and
Effectiveness of Mineral Admixtures in Preventing Excessive Expansion of Concrete Due to the Alkali-Aggregate Reaction (**C 441 – 59 T**).

Tentative Specifications for:

Raw or Calcined Natural Pozzolans for Use as Admixtures in Portland Cement Concrete (**C 402 – 58 T**).

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Standard Method of Test for:

Cement Content of Hardened Portland Cement Concrete (**C 85 – 54**).

Standard Method of:

Measuring Length of Drilled Concrete Cores (**C 174 – 49**).

Recommended Practice for:

Petrographic Examination of Aggregates for Concrete (**C 295 – 54**), with a note at the end of Section 1 to read as follows:

NOTE.—It is recommended that the rock and mineral names in Descriptive Nomenclature of Constituents of Natural Mineral Aggregates (ASTM Designation: C 294) be used insofar as they are appropriate in reports prepared according to this recommended practice.

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following tentatives:

Tentative Method of Test for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar Bar Method) (C 227 – 60 T):[5]

Section 4(b).—Change the last sentence to read: "The sieve splits shall then be dried and recombined in the quantity required for one batch of mortar to the grading prescribed in Table II."

Section 7.—In the third sentence, change "0.01 per cent" to "0.001 per cent" and change the word "report" to "record."

Change the last sentence of Section 7 to read as follows:

> Report the average of the expansions of the four specimens of a given cement-aggregate combination to the nearest 0.01 per cent as the expansion for the combination at a given period.

Section 10.—Change the word "reproducibility" to "repeatability" in the heading and in lines 1 and 8. In line 7 change the word "exceptions" to "exception" and the word "if" immediately following "exceptions" to "when."

Tentative Method of Test for Potential Volume Change of Cement-Aggregate Combinations (C 342 – 55 T):[2]

Section 7.—In the first sentence, change "0.01 per cent" to "0.001 per cent" and change the word "report" to "record."

Change the last sentence of Section 7 to read as follows:

Report the average of the expansions of the three specimens of a given cement-aggregate combination to the nearest 0.01 per cent as the expansion for the combination at a given period.

Section 8.—Change the word "reproducibility" to "repeatability" in the heading and in lines 1 and 9.

Tentative Method of Test for Ball Penetration in Fresh Portland Cement Concrete (C 360 – 55 T):[2]

Section 3.—Change the last sentence of Section 3 to read as follows:

The minimum horizontal distance from the center line of the handle to the nearest edge of the level surface on which the test is to be made shall be 9 in.

Section 4.—In the third sentence, delete the words "and the ball."

Insert the following new sentence after the third sentence to read: "Lower the weight to the surface of the concrete and release slowly."

Tentative Method of Test for Effectiveness of Mineral Admixtures in Preventing Excessive Expansion of Concrete Due to Alkali-Aggregate Reaction (C 441 – 59 T):[4]

Section 3(a).—Change the last sentence to read: "The sieve splits shall then be recombined in the quantity required for one batch of mortar in the grading as follows:" (Retain tabulation).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[7]

This report has been submitted to letter ballot of the committee, which consists of 191 active members commanding 140 votes plus 9 honorary members having voting privileges; 171 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

Bryant Mather,
Chairman.

Paul Klieger,
Secretary.

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[7] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-11

ON

GYPSUM*

Committee C-11 on Gypsum held two meetings during the year: in Atlantic City, N. J., on June 30 and July 1, 1960, and in Cincinnati, Ohio, on Feb. 2 and 3, 1961.

The committee consists of 31 members, of whom 30 are voting members; 12 are classified as producers, 9 as consumers, 9 as general interest members, with one consulting member.

Henry Omson was elected secretary due to the resignation of R. H. Faber; and C. A. Immer was elected to the Executive Committee due to the death of T. I. Coe.

NEW STANDARDS

The methods listed below currently appear in the Standard Methods of Testing Gypsum and Gypsum Products (C 26 – 60).[1] The committee recommends that these methods be published as separate standards. The sections of Methods C 26 where these tests currently appear are indicated below; the methods themselves are not appended to this report. It has been necessary to make minor changes in the methods as indicated below in order to complete them for publication as separate standards.

Standard Methods of Chemical Analysis of Gypsum and Gypsum Products:

New Section 1.—Add the following new Section 1 on scope:

1. *Scope.*—(*a*) These methods cover procedures for the chemical analysis of gypsum and gypsum products.

(*b*) The test procedures appear in the following order:

	Sections
Free Water	2
Preparation of Sample	3
Complete Procedure	4 to 16
Determination of Sand in Set Plaster	17 to 20
Wood-Fiber Content in Wood-Fiber Gypsum Plaster	21

Section 2.—Retain present Section 2(*a*) of Methods C 26 and revise Paragraph (*b*) to read "(*b*) Retain the sample for chemical analysis."

Section 3.—Add a new Section 3 to read as follows, retaining the present Footnote 3 referenced to the word "sieve:"

3. *Preparation of Sample.*—Prepare a dried sample as described in Section 2. Reduce the sample to pass a No. 60 (250-μ) sieve[3] taking extreme care not to expose unduly the material to moisture or to overheating. Thoroughly remix the ground sample, and store it in an airtight container until used.

Sections 4 to 21.—Renumber present Sections 5 to 17 and Sections 30 to 34 of Methods C 26 as Sections 4 to 16 and

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] 1960 Supplement to Book of ASTM Standards, Part 4.

Sections 17 to 21 respectively, changing section references where necessary.

Standard Methods of Physical Testing of Gypsum Plasters and Gypsum Concrete:

New Section 1.—Add the following new Section 1 on scope:

1. *Scope.*—(*a*) These methods cover procedures for the physical testing of gypsum plasters and gypsum concrete.

(*b*) The test procedures appear in the following order:

	Sections
Free Water	2
Fineness	3
Precaution for Physical Tests	4
Normal Consistency of Gypsum Plaster	5 and 6
Normal Consistency of Gypsum Concrete	7 and 8
Time of Setting	9 and 10
Compressive Strength	11 to 14
Density	15

Sections 2 and 3.—Retain present Sections 2 and 3 from Methods C 26.

Sections 4 to 15.—Renumber present Sections 18 to 29 of Methods C 26 as Sections 4 to 15, including the three present illustrations.

Appendix.—Add the present Appendix from Methods C 26.

Standard Methods of Physical Testing of Gypsum Board Products and Gypsum Partition Tile or Block:

New Section 1.—Add the following new Section 1 on scope:

1. *Scope.*—(*a*) These methods cover procedures for the physical testing of gypsum board products (including gypsum lath) and gypsum partition tile or block.

(*b*) The test procedures appear in the following order:

	Sections
Compressive Strength of Gypsum Partition Tile or Block	2 and 3
Flexural Strength of Gypsum Boards	4 to 6
Weight and Thickness of Paper Surfacing of Gypsum Boards	7 and 8
Flexural Strength of Precast Reinforced Gypsum Slabs	9 and 10
Nail Retention of Precast Reinforced Gypsum Slabs	11 and 12
Determination of Edge Tolerance of Recessed or Tapered-Edge Gypsum Wallboard	13 to 15
Water Resistance Characteristics of Core-Treated Water-Repellent Gypsum Sheathing	16 and 17

Sections 2 to 17.—Renumber present Sections 35 to 50 of Methods C 26 as Sections 2 to 17.

NEW TENTATIVES

The committee recommends for publication as tentative the following specification and methods as appended hereto:[2]

Tentative Specification for:

Joint Treatment Materials for Gypsum Wallboard Construction.

Tentative Methods of Testing:

Joint Treatment Materials for Gypsum Wallboard Construction.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Specifications for Annular Ringed Nails for Gypsum Wallboard (C 380 - 60 T):[3]

Section 2.—Change the first sentence to read "Steel wire used in the manufacture of nails shall be of hard-drawn low- or medium-low-carbon steel, entirely suitable for the purpose intended."

Section 3(*e*).—Change to read: "(*e*) Distance from the top of the head to the first ring shall be equal to the nominal thickness of the gypsum wallboard for which the nail is specified, with permissible tolerance of $\pm\frac{1}{32}$ in."

Tentative Specifications for Gypsum Backing Board (C 442 - 59 T):[4]

Section 1.—Add the following to the

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[2] The new tentatives appear in the 1961 Book of ASTM Standards, Part 4.

[3] 1958 Book of ASTM Standards, Part 4.

[4] 1959 Supplement to Book of ASTM Standards, Part 4.

end of the first sentence "designed to be used as a base layer or backing material in multi-layer construction."

Section 2.—Delete the present last two sentences, and add the following:

> Type X (special fire retardant) designates gypsum backing board complying with these specifications that provides at least 1-hr fire retardant ratings for boards ⅝ in. thick, or ¾-hr fire retardant ratings for boards ½ in. thick applied in single layer nailed application on each face of load-bearing wood framing members when tested in accordance with the requirements of the Methods of Fire Tests of Building Constructions and Materials (ASTM Designation: E 119).
>
> NOTE.—Consult manufacturers for test data on assembly particulars, materials, and ratings for other types of constructions.

Section 3.—Add to the table in this section a thickness of 1 in. with a load under bearing edges across fiber of surfacing of 225 lb, and a load under bearing edges parallel to fiber of surfacing of 90 lb.

Section 4.—In Paragraph (*a*), add to the end of the first sentence "or shaped for specific construction systems."

In Paragraph (*b*), add a nominal thickness of 1 in.

In Paragraph (*c*), add to the end of the paragraph, "Other widths are permissible, as required by the specific construction systems."

In Paragraph (*e*), add to the table a thickness of 1 in. with a minimum weight of 3600 lb, and a maximum weight of 4800 lb.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revision as follows of the Standard Specifications for Gypsum Wallboard (**C 36– 60**),[3] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revision may be referred to letter ballot of the Society:

Section 5(*a*).—In the last sentence change "0.010 in." to read "0.015 in."

WITHDRAWAL OF STANDARD

The committee recommends the withdrawal of the Standard Methods of Testing Gypsum and Gypsum Products (**C 26 – 60**). These methods are replaced by the three new Standard Methods noted earlier in this report.

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Standard Specifications for:
Gypsum (**C 22 – 50**),
Gypsum Lath (**C 37 – 54**),
Gypsum Partition Tile or Block (**C 52 – 54**),
Gypsum Molding Plaster (**C 59 – 50**),
Keene's Cement (**C 61 – 50**),
Gypsum Sheathing Board (**C 79 – 54**),
Gypsum Concrete (**C 317 – 55**), and
Gypsum Formboard (**C 318 – 55**).

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Specifications for Precast Reinforced Gypsum Slabs (**C 377 – 58 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

This report has been submitted to letter ballot of the committee, which consists of 30 voting members; 24 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

G. W. JOSEPHSON,
Chairman.

H. OMSON,
Secretary.

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[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-12
ON
MORTARS FOR UNIT MASONRY*

Committee C-12 on Mortars for Unit Masonry held two meetings during the past year: on June 27, 1960, in Atlantic City, N. J.; and on January 31, 1961, in Cincinnati, Ohio.

The committee consists of 54 members, of whom 22 are classified as producers, 16 as consumers, 15 as general interest members, and 1 consulting member.

Two esteemed long-time members of the committee, Theodore Irving Coe and Walter C. Voss, died during the past year.

NEW TENTATIVE

The committee recommends for publication as tentative the proposed Tentative Specifications for Mortar and Grout for Reinforced Masonry, as appended hereto.[1]

REVISION OF TENTATIVE

The committee recommends revisions as follows of the Tentative Specifications for Mortar for Unit Masonry (**C 270 - 59 T**)[2] and continuation of the specifications as tentative:

Section 2(a).—After "*Slag Cement*" insert the following phrase "(for use in property specifications only)."

Section 2(e).—Add a note to read as follows:

> Calcium chloride when provided for in the contract specifications may be used as an accelerator in amounts not exceeding 2 per cent by weight of the portland cement content of the mortar.

Section 2(f).—Delete the last sentence.

Section 3(a).—Add to the table in Note 1: "Portland blast-furnace slag cement . . . 94 (approx)."

Section 3(b).—Change to read as follows:

> All cementitious materials and aggregate shall be mixed for at least 3 min with the maximum amount of water to produce a workable consistency in a mechanical batch mixer (Note 2).

Retain the present Note 2.

New Section 3(c).—Add a new Section 3(*c*) to read as follows:

> Mortars that have stiffened because of evaporation of water from the mortar shall be retempered by adding water as frequently as needed to restore the required consistency. Mortars shall be used and placed in final position within 2½ hr after mixing.

Section 4.—Letter the present Section 4 as Paragraph (*a*), and add a new Paragraph (*b*) to read as follows:

> No change in the proportions established for mortar accepted under the property specifications shall be made nor shall materials with different physical characteristics be utilized in mortar used in the work unless compliance with the requirements of the property specifications is reestablished.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Specifications for Aggregate for Masonry Grout (**C 404 – 57 T**)[3] be approved for reference to letter ballot of the Society for adoption as standard without revision.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] The new tentative appears in the 1961 Book of ASTM Standards, Part 4.

[2] 1959 Supplement to Book of ASTM Standards, Part 4.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Research and Methods of Test (P. L. Rogers, chairman) has completed an extensive series of tests on the effect of a waiting period in mixing of mortar prior to testing. Insufficient effect was observed to merit a change in test methods.

Subcommittee III on Specifications (P. V. Johnson, chairman) has completed a proposed Tentative Specification for Mortar and Grout for Reinforced Masonry, which is appended to this report.[1]

Subcommittee IV on Aggregates (D. L. Bloem, chairman) has been revising the sand grading requirements of Specifications C 144 after completion of a comprehensive test series to determine the effect of sand grading on the properties of masonry mortars.

This report has been submitted to letter ballot of the committee, which consists of 54 members; 53 returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. C. PLUMMER,
Chairman.

C. U. PIERSON, JR.,
Secretary.

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[3] 1958 Book of ASTM Standards, Part 4.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-13

ON

CONCRETE PIPE*

Committee C-13 on Concrete Pipe held one meeting during the year: in Chicago, Ill. on Oct. 19, 1960.

The committee consists of 44 members, of whom 20 are classified as producers, 12 as consumers, and 12 as general interest members.

NEW TENTATIVES

The committee recommends for publication as tentative the following specifications, as appended hereto:[1]

Tentative Specifications for Precast Reinforced Concrete Manhole Risers and Tops:

This tentative is the result of a study of related data and consideration of recommendations submitted to the committee by the Technical Problems Committee of the American Concrete Pipe Assn.

Tentative Specifications for Cast-In-Place Non-Reinforced Concrete Irrigation Closed Conduit:

This tentative is the result of considerable study of related data.

REVISION OF TENTATIVE

The committee recommends revisions as follows of the Tentative Specifications for Reinforced-Concrete Culvert, Storm Drain, and Sewer Pipe (**C 76 - 60 T**):[2]

Table II.—Add a Wall C column as shown in the accompanying Table I.

Table III.—Add a Wall C column as shown in the accompanying Table II.

Table IV.—Extend internal diameters of pipe up to and including 108 in. for Walls A, B, and C. Do not add add steel areas or wall thicknesses but reference a new Footnote *b*.

Add a new Footnote *b* to read as follows:

> The manufacturer shall submit wall thickness, steel design, and concrete strength to the purchaser for approval prior to manufacture."

Table V.—Add a Wall A column with internal diameters of pipe from 12 in. through 108 in. Do not add steel areas or wall thicknesses but reference a new Footnote *b*.

For Wall B and C, extend internal diameters of pipe up to and including 108 in. Do not add steel areas or wall thicknesses but reference a new Footnote *b*.

Add a new Footnote *b* to read the same as the new Footnote *b* to Table IV.

Section 22(*b*).—After the words "pipe tested" in the seventh line, add the following parenthetical phrase "(including preliminary tests)."

Section 25(*d*).—After the words "lay-

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] The new tentatives appear in the 1961 Book of ASTM Standards, Part 5.

[2] 1960 Supplement to Book of ASTM Standards, Part 5.

TABLE I.—DESIGN REQUIREMENTS FOR CLASS II REINFORCED CONCRETE PIPE.

(Additions to Table II of Specifications C 76)

Internal Diameter of Pipe, in.	Wall C				
	Concrete Strength, 4000 psi				
	Minimum Reinforcement, sq in. per foot of pipe barrel				
	Minimum Wall Thickness, in.	Circular Reinforcement		Elliptical Reinforcement	
		Inner Cage	Outer Cage		
36	4¾	0.07	0.07	0.08	
42	5¼	0.10	0.08	0.11	
48	5¾	0.14	0.11	0.15	
54	6¼	0.17	0.13	0.19	
60	6¾	0.22	0.17	0.24	
66	7¼	0.25	0.19	0.28	
72	7¾	0.30	0.23	0.33	
78	8¼	0.35	0.26	0.39	
84	8¾	0.41	0.31	0.46	
90	9¼	0.48	0.36	0.53	
96	9¾	0.55	0.41	0.61	
	Concrete Strength, 5000 psi				
102	10¼	0.62	0.47	Inner Circular Plus Elliptical	0.22 0.47
108	10¾	0.70	0.53	Inner Circular Plus Elliptical	0.25 0.53

TABLE II.—DESIGN REQUIREMENTS FOR CLASS III REINFORCED CONCRETE PIPE.

(Additions to Table III of Specifications C 76)

Internal Diameter of Pipe, in.	Wall C				
	Concrete Strength, 4000 psi				
	Minimum Reinforcement, sq in. per foot of pipe barrel				
	Minimum Wall Thickness	Circular Reinforcement		Elliptical Reinforcement	
		Inner Cage	Outer Cage		
36	4¾	0.08	0.07	0.09	
42	5¼	0.12	0.09	0.13	
48	5¾	0.16	0.12	0.18	
54	6¼	0.21	0.16	0.23	
60	6¾	0.25	0.19	0.28	
66	7¼	0.31	0.23	0.34	
72	7¾	0.36	0.27	0.40	
78	7¼	0.42	0.32	0.47	
84	8¾	0.50	0.38	0.56	
	Concrete Strength, 5000 psi				
90	9¼	0.59	0.45	0.66	
96	9¾	0.70	0.53	Inner Circular Plus Elliptical	0.25 0.53
102	10¼	0.83	0.62	Inner Circular Plus Elliptical	0.30 0.62
108	10¾	0.99	0.74	Inner Circular Plus Elliptical	0.36 0.74

ing lengths" in the first line, add the following parenthetical phrase: "(See "L" in Figs. 2 and 3)."

ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that the Tentative Specifications for Reinforced Concrete Low-Head Pressure Pipe (**C 361 - 59 T**)[3] be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

Section 13(*b*).—Change Item 6 to read as follows:

> (6) *Accelerated Aging.*—To be conditioned in a circulating hot air oven 96 hr at 158 F with the tensile strength loss of 15 per cent maximum, and elongation loss of 20 per cent maximum of the original.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

This report has been submitted to letter ballot of the committee which consists of 44 voting members; 44 members returned their ballots, of whom 41 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

R. R. LITEHISER,
Chairman.

HOWARD F. PECKWORTH,
Secretary.

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[3] 1959 Supplement to Book of ASTM Standards, Part 5.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-14
ON
GLASS AND GLASS PRODUCTS*

Since its last report to the Society in June, 1958, Committee C-14 on Glass and Glass Products has held six meetings: on October 16, 1958, in Bedford Springs, Pa.; May 20, 1959, in Chicago, Ill.; September 17, 1959, in New York City; April 27, 1960, in Philadelphia, Pa.; October 13, 1960, in Bedford Springs, Pa.; and April 24, 1961, in Toronto, Ont.

The committee consists of 51 voting members of whom 18 are classified as producers, 14 as consumers, and 19 as general interest members.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

In addition to the recommendations of Committee C-14 which were accepted by the Society through the Administrative Committee on Standards during 1958 and 1959, and were announced at the time in the ASTM BULLETIN, the Administrative Committee accepted on May 10, 1961, the recommendation of the committee for the tentative revision of the Standard Definitions of Terms Relating to Glass and Glass Products (C 162 - 56). The revised definitions are available as a separate reprint.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as appended hereto[1] of the Standard Methods of Testing Structural Non-Load-Bearing Cellular Glass Blocks (**C 240 - 56**),[2] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

The recommendation in this report has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Definitions (H. H. Holscher, chairman).—Forty new and revised definitions were developed for inclusion in Definitions C 162.

Subcommittee II on Chemical Analysis (F. L. White, chairman).—An interlaboratory study on additional chemical analysis of glass sand has been completed. Refinement of the procedure was indicated and a new interlaboratory test is being developed.

The first interlaboratory test of the analysis of soda lime glass was completed and the resulting data reviewed.

Subcommittee III on Chemical Properties (J. F. Greene, chairman) is working closely with Subcommittee VII A on durability testing. The committee is attempting to develop data from the present test procedure in order to determine the lack of reproducibility of durability tests.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] The revised standard appears in the 1961 Book of ASTM Standards, Part 5.

[2] 1958 Book of ASTM Standards, Part 5.

[3] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee IV on Physical and Mechanical Properties (C. L. Babcock, chairman).—The Method of Test for Annealing Point and Strain Point of Glass (C 336) is being critically reviewed for possible changes before its adoption as a standard method.

A member of this subcommittee is working with Committee E-1 on Methods of Testing in the development of general ASTM standards for measuring the linear and cubical thermal expansion of materials.

Subcommittee VI on Glass Construction Block and Tile (A. H. Baker, chairman) has completed the revision of Method C 240 referred to earlier in this report.

Subcommittee VII on Glass Containers (R. E. Mould, chairman).—It was announced that a new set of strain disks used in conjunction with the polariscopic examination, Method C 148, are available from the British Glass Industry Research Assn., Sheffield, England.

Section "A," Pharmaceutical Containers, W. R. Prindle, chairman, has reviewed a boiling acid test sponsored by the International Standards Organization. The interlaboratory data collected using this test have been submitted to the committee responsible for this test. This section has also cooperated with the committee for the publication of the new edition of the U. S. Pharmacopoeia in regard to details of the glass container durability test methods.

Subcommittee VIII on Flat Glass (H. R. Swift, chairman) is in correspondence with the Swedish Glasinstitutet regarding questions on standard tests for mirrors.

This report has been submitted to letter ballot of the committee, which consists of 51 members; 23 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

L. G. Ghering,
Chairman.

F. V. Tooley,
Secretary.

REPORT OF COMMITTEE C-15
ON
MANUFACTURED MASONRY UNITS*

Committee C-15 on Manufactured Masonry Units held two meetings during the year: on July 1, 1960, in Atlantic City, N. J., in conjunction with the Annual Meeting of the Society, and on Feb. 2, 1961, in Cincinnati, Ohio, during ASTM Committee Week.

The committee consists of 72 members, of whom 36 are classified as producers, 17 as consumers, and 19 as general interest members.

During the past year, members of the committee were saddened to learn of the death of two of its most esteemed members of long standing: T. I. Coe, Honorary Member, and for many years First Vice-Chairman of the committee, died on Nov. 12, 1960; C. C. Connor died on March 4, 1961.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1960 Annual Meeting, Committee C-15 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Revision of Standard Specifications for:

Building Brick (62 – 58).

Revision of Tentative Specifications for:

Ceramic Glazed Structural Clay Facing Tile, Facing Brick, and Solid Masonry Units (**C 126 – 59 T**).

These recommendations were accepted by the Standards Committee on September 29, 1960, and they appear in the 1960 Supplement to the Book of ASTM Standards, Part 5.

REVISION OF TENTATIVE

The committee recommends the revision as follows of the Tentative Specifications for Ceramic Glazed Structural Clay Facing Tile, Facing Brick, and Solid Masonry Units (**C 126 – 60 T**):[1]

Section 16.—Change paragraph (*c*) to read as follows:

(*c*) *Chemical Resistance Test.*—An end portion of two whole specimens shall be submerged with the glazed surface exposed to a minimum depth of 1½ in. in a 10 per cent solution of HCl for 3 hr. The opposite end portions of the glazed surfaces of the same specimens shall then be similarly submerged in a 10 per cent solution of KOH for 3 hr. These solutions shall be maintained at a temperature of 60 to 80 F (15 to 27 C). The finishes shall then be rinsed, dried, and examined for changes of texture and color, if any.

NOTE.—A 10 per cent solution of HCl is prepared by volume by using 10 per cent concentrated HCl which is 12 *N* or 37.0 per cent. A 10 per cent solution of KOH is prepared by weight using, for example, 10 g of KOH diluted to 100 g solution. Distilled water is used and fresh solutions should be made for each test set up.

In Paragraph (*d*) delete the parenthetical phrase "(not exceeding 120 F)" in lines 37 and 38.

The recommendation in this report has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Clay Brick and Structural Clay Tile (J. E. Neville, chairman) has continued its study of problems such as chippage, dimensional tolerances, and durability of surface coloring of facing brick. This work is being carried on through task groups who report periodically to the subcommittee. A program investigating an accelerated freezing-and-thawing test is also being carried on under the sponsorship of the subcommittee.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] 1958 Book of ASTM Standards, Part 5.

[2] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee III on Concrete and Sand Lime Units (R. E. Copeland, chairman) is considering a program of tests to investigate the need for capping the bearing surfaces of concrete brick compressive strength specimens. This program was suggested in view of comparative strength values reported for capped and uncapped brick in tests conducted by a member.

Subcommittee V on Water-Retardant Materials for Masonry Units (C. C. Fishburn, chairman) has continued through task groups its investigative work, including cooperative tests, which should provide the information needed for the development of specifications and methods of test for water retardant materials.

Subcommittee VI on Glazed Brick and Tile (E. F. Wanner, chairman) has reviewed a number of editorial revisions of Tentative Specifications for Ceramic Glazed Structural Clay Facing Tile, Facing Brick, and Solid Masonry Units (C 126) which had been recommended, as well as several desirable revisions in substance of this tentative. The subcommittee has under consideration a request for development of specifications for ceramic glazed units suitable for exterior use.

Subcommittee IX on Chemical-Resistant Units (W. W. Perkins, chairman).—A proposed specification for chemical-resistant ceramic tower packings is nearing completion. It is hoped that the final draft of this proposed specification will be ready for presentation to Committee C-15 at the Annual Meeting for approval by the committee.

Subcommittee X on Clay Drain Tile (J. C. Sutton, chairman) is considering a number of revisions of Tentative Specifications for Clay Drain Tile (C 4) which had been recommended as the result of field experience in the use of this specification. Subcommittee X is also preparing a new specification for perforated clay drain tile.

Subcommittee XI, Editorial (Frank Biberstein, chairman) has continued its work on reviewing all standards, tentatives, and proposed tentatives under the jurisdiction of Committee C-15. Recommended editorial revisions of these documents were made to the appropriate technical subcommittees for action.

This report has been submitted to letter ballot of the committee, which consists of 72 members; 55 members returned their ballots, of whom 54 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. W. WHITTEMORE,
Chairman.

M. H. ALLEN,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-15 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

Ceramic Glazed Structural Clay Facing Tile, Facing Brick, and Solid Masonry Units (C 126 - 60 T), and

Vitrified Clay Filter Block for Trickling Filters (C 159 – 59 T).

Revision of Tentative Method of Test for:

Drying Shrinkage of Concrete Block (C 426 – 58 T).

These recommendations were accepted by the Standards Committee on September 12, 1961, and the revised tentative specifications and method appear in the 1961 Book of ASTM Standards, Part 5.

REPORT OF COMMITTEE C-16
ON
THERMAL INSULATING MATERIALS*

Committee C-16 on Thermal Insulating Materials and its subcommittees held two meetings during the year: at The Hotel Marcy, Lake Placid, N. Y. on September 25 to 28, 1960, and at the Hotel Netherland Plaza, Cincinnati, Ohio in conjunction with Committee Week on January 29 to February 1, 1961.

At the Lake Placid meeting, Russell H. Heilman, who has the distinction of having attended all the meetings of Committee C-16 since its inception, was honored with the presentation of a framed scroll citing him for his meritorious service and contributions to the committee. Mr. Heilman has the honor of being the recipient of the Society's Award of Merit.

The committee consists of 103 voting members, of whom 50 are classified as producers, 28 as consumers, and 25 as general interest members. There are also 28 non-voting members, 3 associate members, and 5 honorary members.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1960 Annual Meeting, Committee C-16 presented to the Society through the Administrative Committee on Standards the following recommendations which were accepted on the dates indicated:

New Tentative Methods of Test for:

Mastic Coatings Used in Conjunction with Insulations (C 461 – 60 T) (Accepted Sept. 29, 1960), and

Corrosion of Steel in Contact with Wet Thermal Insulating Cement (C 464 – 61 T) (Accepted March 1, 1961).

Revision of Tentative Specifications for:

Mineral Fiber Blanket and Felt Insulation (Industrial Type) for Low Temperature (C 264 – 55 T) (Accepted Sept. 29, 1960).

Revision and Reversion to Tentative of Standard Specifications for:

Mineral Fiber Thermal Insulating Cement (C 195 – 48) (Accepted Sept. 29, 1960), and

Asbestos Thermal Insulating and Finishing Cement (C 194 – 48) (Accepted Jan. 3, 1961).

The new Tentative Method C 461 and the revised Tentative Specifications C 264 and C 195 appear in the 1960 Supplement to Book of ASTM Standards, Part 5. The new Tentative Method C 464 and the revised Tentative Specification C 194 are available as separate reprints.

TENTATIVE REVISION OF STANDARD

The committee recommends tentative revisions as follows of the Standard Definitions of Terms Relating to Thermal Insulating Materials (**C 168 – 56**).[1]

New Definitions.—Add the following new definitions:

Blanket Thermal Insulation.—A flexible thermal insulation that can readily be conformed to curved or irregular surfaces, suitably bonded together to provide units of substantial area for handling and application and which may be faced or reinforced with confining media.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] 1958 Book of ASTM Standards, Part 5.

Wood Fiber Blanket Thermal Insulation.—A blanket thermal insulation composed of wood fibers which may have been chemically treated and with or without added binders.

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends revisions as appended hereto[2] of the Standard Methods of Sampling and Mixing Thermal Insulating Cement (**C 163 – 44**)[1] and reversion of the methods to tentative.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for:

Expanded or Exfoliated Vermiculite Thermal Insulating Cement (**C 196 – 59 T**),[3] and
Diatomaceous Silica Thermal Insulating Cement (**C 197 – 59 T**).[3]

Tentative Methods of Test for:

Mean Specific Heat of Thermal Insulation (**C 351 – 59 T**),[3]
Hot Surface Performance of High-Temperature Thermal Insulations (**C 411 – 58 T**),[1]
Weight Loss by Tumbling of Preformed Thermal Insulation (**C 421 – 58 T**),[1] and
Normal Total Emittance of Surfaces of Materials 0.01 in. or Less in Thickness at Approximately Room Temperature (**C 445 – 59 T**).[3]

REVISION OF TENTATIVE

The committee recommends that the Tentative Specifications for Mineral Fiber Blanket and Felt Insulation (Industrial Type) for Elevated Temperatures (**C 382 – 56 T**)[1] be revised as appended hereto[4] and continued as tentative.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revision as follows of the Standard Method of Test for Covering Capacity and Volume Change upon Drying of Thermal Insulating Cement (**C 166 – 52**)[1] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Section 3(a).—Change the inside dimensions of the mold from "1 by 12 by 36 in." to read "1 by 8 by 36 in."

WITHDRAWAL OF STANDARD

The committee recommends the withdrawal of the Standard Method of Test for Storage Density of Thermal Insulating Cement (**C 164 – 52**).[1]

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Standard Specifications for:

85 per cent Magnesia Block-Type Thermal Insulation (**C 319 – 55**), and
85 per cent Magnesia Molded-Type Thermal Insulation for Pipes (**C 320 – 55**).

Standard Methods of Test for:

Compressive Strength of Preformed Block-Type Thermal Insulation (**C 165 – 54**), and
Thermal Conductivity of Materials by Means of the Guarded Hot Plate (**C 177 – 45**).

Recommended Practice for:

Clearance of Preformed Thermal Pipe Insulation (**C 312 – 55**).

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[2] The revised methods appear in the 1961 Book of ASTM Standards, Part 5.

[3] 1959 Supplement to Book of ASTM Standards, Part 5.

[4] The revised specifications appear in the 1961 Book of ASTM Standards, Part 5.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Specifications for:

Diatomaceous Earth Block-Type Thermal Insulation (**C 333 – 59 T**),
Diatomaceous Earth Thermal Insulation for Pipes (**C 334 – 59 T**),
Amosite Asbestos Thermal Insulation for Pipes (**C 391 – 59 T**), and
85 per cent Magnesia Thermal Insulating Cement (**C 193 – 59 T**).

Tentative Methods of Test for:

Thermal Conductivity of Pipe Insulation (**C 335 – 54 T**),
Thermal Conductivity of Insulating Materials at Low Temperature by Means of the Wilkes Calorimeter (**C 420 – 58 T**),
Water Vapor Transmission of Materials Used in Building Construction (**C 355 – 59 T**), and
Breaking Strength and Calculated Modulus of Rupture of Preformed Insulation for Pipes (**C 446 – 59 *T***).

Tentative Method for Determining:

Maximum Use Temperature of Preformed High-Temperature Insulation (**C 447 – 59 T**).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee S-VIII on General Standards (H. P. Hoopes, chairman) expects to present a modification of the Recommended Practices for Prefabrication and Field Fabrication of Thermal Insulation Fitting Covers (C 450 – 60 T) to include the cutting of beveled blocks. This will eliminate the need for writing a new standard.

Subcommittee S-XI on Reflective Insulation (G. E. Gronemeyer, chairman) proposed a research project on the effectiveness of reflective insulation in gaseous environments of high temperatures and high pressures to determine the values of reflective insulations that may be used in nuclear reactors. The West Virginia University submitted a proposal to the committee for the undertaking of this project. The main committee indicated that this did not have sufficient value at this time for the thermal insulation industry as a whole to warrant the expense.

Task Force on Philosophy of Specifications has the responsibility of preparing a plan for including specifications for materials with more usable values to benefit the users of these standards.

This report has been submitted to letter ballot of the committee, which consists of 103 voting members; 78 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. C. LEWIS,
Chairman.

J. M. HIGH,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-16 presented to the Society through the Administrative Committee on Standards the recommendation for publication as tentative of the Method of Test for Resistance to Dropping of Preformed Block-Type Thermal Insulation. This recommendation was accepted by the Standards Committee on January 3, 1962, and the method is available as a separate reprint bearing the designation C 487 – 62 T.

REPORT OF COMMITTEE C-17
ON
ASBESTOS-CEMENT PRODUCTS*

Committee C-17 on Asbestos-Cement Products and its subcommittees met on June 27 and 28, 1960, in Atlantic City, N. J. during the Annual Meeting of the Society, and on November 30 and December 1, 1960, in Buffalo, N. Y.

The committee consists of 38 members, of whom 32 are voting members; 10 are classified as producers; 6 as consumers; 16 as general interest members; and 1 consulting member.

During the year the committee experienced a loss through the death of one of its esteemed members, Theodore I. Coe.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Specifications and Methods of Test for Corrugated Asbestos-Cement Sheets (**C 221 - 58 T**)[1] be approved for reference to letter ballot of the Society for adoption as standard without revision.

ADOPTION OF TENTATIVE REVISION AS STANDARD

The committee recommends that the tentative revisions of Tables I, II, and III of the Standard Specifications for Flat Asbestos-Cement Sheets (**C 220 - 60**)[2] be approved for reference to letter ballot of the Society for adoption as standard.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Research (William Lerch, chairman) prepared a program of papers on the durability of asbestos-cement products for the committee meeting during the 1961 Annual Meeting in Atlantic City, N. J.

Subcommittee III, ISO/TC 77 on Asbestos-Cement Products (J. R. Cran, chairman).—R. F. Orth of the Johns-Manville Corp. and A. E. Binger of The Phillip Carey Manufacturing Co., have been designated as the U. S. delegates to the meeting of ISO/TC 77 to be held in London, June 19 to 23, 1961.

Subcommittee IV on Roofing and Siding (F. A. Crippen, chairman) reviewed the third draft proposal for ISO Recommendations on Asbestos-Cement Slates for Roofing and Cladding. With no clear understanding of the word "cladding" or of foreign application techniques, it was decided that this problem be referred to the American Standards Association for clarification. The Swedish comments on the first draft proposal for an ISO Recommendation on Asbestos-Cement Siding have been discussed. The subcommittee agreed to send the latest revision of the ASTM Specification for Asbestos-Cement Siding (C 223) to the ISO as the comments from the United States on draft proposals for asbestos-cement siding recommendations.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] 1958 Book of ASTM Standards, Part 5.

[2] 1960 Supplement to Book of ASTM Standards, Part 5.

A task force composed of F. V. Camarda, chairman, R. S. Miller, and E. A. Farrell was appointed to study the ISO methods for impermeability tests, frost cracking tests, and the Canadian methods for color stability for consideration for inclusion of these tests in ASTM specifications.

Subcommittee V on Corrugated and Flat Sheets (S. E. Williams, chairman) has under consideration the development of specifications and methods of tests for panel board consisting of asbestos-cement faces and organic-fiber cores. A task force composed of W. A. Heausler, chairman, H. W. Greider, and C. V. French, has been given this assignment.

Subcommittee VI on Asbestos-Cement Pipe (C. R. Meek, chairman).—The subcommittee prepared, with the aid of Subcommittee II on Research, a method of test for determination of uncombined calcium hydroxide.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

This report has been submitted to letter ballot of the committee, which consists of 32 voting members; 20 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. V. FRIEDLAENDER,
Chairman.

SIMON COLLIER,
Secretary.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-18
ON
NATURAL BUILDING STONES*

Committee C-18 on Natural Building Stones held one meeting during the year: on May 26, 1961, in Washington, D. C.

The committee consists of 21 members, of whom 8 are classified as producers, 7 as consumers, and 6 as general interest members. There are 3 consulting members.

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Standard Methods of Test for:

Absorption and Bulk Specific Gravity of Natural Building Stone (**C 97 – 47**),
Modulus of Rupture of Natural Building Stone (**C 99 – 52**),
Water Absorption of Slate (**C 121 – 48**),
Compressive Strength of Natural Building Stone (**C 170 – 50**), and
Abrasion Resistance of Stone Subjected to Foot Traffic (**C 241 – 51**).

Standard Methods of:

Flexure Testing of Slate (Modulus of Rupture, Modulus of Elasticity) (**C 120 – 52**).

Standard Definitions of Terms Relating to:

Natural Building Stones (**C 119 – 50**).

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Method of Test for Combined Effect of Temperature Cycles and Weak Salt Solutions on Natural Building Stone (**C 218 – 48 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[1]

SUBCOMMITTEE ACTIVITIES

Subcommittee II on Nomenclature and Definitions (L. W. Currier, chairman) has been asked to give attention to definitions relating to texture and for better classification in cut stone.

Subcommittee IV on Specifications (F. S. Eaton, chairman).—Four task groups are actively studying proposed specifications for slate, limestone, sandstone, and marble respectively. A fifth task group on granite is reviewing the Tentative Specification for Structural Granite (C 422 – 58 T).

This report has been submitted to letter ballot of the committee, which consists of 21 members; 16 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

F. S. EATON,
Acting Chairman.

W. F. MULLEN,
Acting Secretary.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-19

ON

STRUCTURAL SANDWICH CONSTRUCTIONS*

Committee C-19 on Structural Sandwich Constructions held two meetings during the year: on Oct. 4 and 5, 1960, in Philadelphia, Pa., and on April 18 and 19, 1961, in St. Paul, Minn.

The committee consists of 57 voting members, of whom 37 are classified as producers, 8 as consumers, 12 as general interest members, and 2 consulting members.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto:[1]

Tentative Methods of Test for:

Flexure-Creep of Sandwich Constructions, and Laboratory Aging of Sandwich Constructions.

REVISION OF TENTATIVE

The committee recommends revisions as follows of the Method of Flatwise Flexure Test of Sandwich Constructions (**C 393 – 57 T**)[2] and continuation of the method as tentative:

Title.—Change to read as follows: "Flexure Test of Flat Sandwich Constructions."

Section 1.—Insert the word "flat" before the words "sandwich constructions" in line 2.

Section 2(b).—Change the word "movements" in the third line to "moments."

Section 7(f).—Change "W_2" to "w_2" in line 2 and in the formula.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Method of Test for Density of Core Materials for Structural Sandwich Constructions (C 271 – 53):[2]

Section 3.—Change to read as follows:

3. The test specimen shall be in the form of a square 3 in. (7.62 cm) in length by 3 in. (7.62 cm) in width by the thickness of the sandwich core material. At least three specimens shall be tested.

Section 5.—Revise to read as follows:

5. Calculate the density in pounds per cubic foot as follows:

$$\text{Density} = \frac{F \times 1728}{L \times W \times T}$$

where:

F = final weight after conditioning, in pounds,
L = length of specimen after conditioning, in inches,
T = thickness of specimen after conditioning, in inches, and
W = width of specimen after conditioning, in inches.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] The new tentatives appear in the 1961 Book of ASTM Standards, Part 5.

[2] 1958 Book of ASTM Standards, Part 5.

If measurements are more conveniently made in the metric system, calculate the density and convert to English units, as follows:

$$\text{Density, lb per cu ft} = \frac{A}{B} \times 62.4$$

where:

A = weight of specimen, in grams, and

B = volume of specimen, in cubic centimeters.

Standard Method of Shear Test in Flatwise Plane of Sandwich Constructions (C 273 – 53):[2]

Title.—Change to read as follows: "Shear Test in Flatwise Plane of Flat Sandwich Constructions or Sandwich Cores."

Section 1.—Add the following sentence at end of the paragraph: "The test may be conducted on core materials in which case facings are not bonded to the core and loading plates are bonded directly to the core."

Section 2.—Change to read as follows:

2. This test provides information on the load-deformation behavior of sandwich constructions or cores when loaded in shear parallel to the plane of the facings. From a complete load-deformation curve it is possible to compute shear stress of the sandwich or core at any load (such as shear stress at proportional limit or at maximum load) and to compute an effective shear modulus (Note 1) of the sandwich as a unit or shear modulus of the core. The shear modulus obtained is that associated with the strains in a plane perpendicular to the facings and parallel to the direction of the applied forces. From tests of sandwich construction it is possible to compute the core shear modulus from the effective sandwich shear modulus if the shear modulus of the facing material is known. By measuring strains in the core, it is possible to obtain core shear modulus directly from a test of a complete sandwich construction. The test does not produce pure shear, but the specimen length is prescribed so that secondary stresses have a minimum effect (Note 2).

Retain the present Note 1. Revise Note 2 to read: "Approximate values of shear properties can be obtained by a flexure test of sandwich construction.[3]" Retain the present Footnote 3.

Section 3(a).—Add the following sentence after the present fourth sentence: "It has been found that loading plates having a stiffness not less than 600,000 psi per inch of width per inch of core thickness have performed satisfactorily."

Section 5.—Change to read as follows:

5. Apply the load to the ends of the rigid plates in compression or tension through a spherical bearing block or a universal joint so as to distribute the load uniformly across the width of the specimen. Apply the load at a constant rate of movement of the movable head of the testing machine (Note) such that the maximum load will occur within 3 to 6 min.

NOTE.—A suggested rate of head movement is 0.005 in. per min per inch of specimen length. For accurate determination of the properties of the specimen, it will be desirable to test under continuously applied load; however, if it is impossible to obtain deformation data, the loading head may be stopped momentarily as readings are taken. The exact procedure used shall be described in the report.

Section 6.—Change the third sentence to read as follows: "Usually 12 readings are adequate to determine the curve to the proportional limit."

Section 7(b).—Change Note 1 to read as follows:

NOTE 1.—If final failure occurs in the bond between the loading plates and the specimen, the test shall be considered unsatisfactory unless the specimen shows severe shear distortion and partial shear failure before final failure.

Section 8.—Add the following new item:

(*5*) Description of specimen failure, whether final failure occurred in core or bond, or in both core and bond. Describe order of failure if possible. Describe appearance of specimen if final failure occurred in bond between specimen and loading plate.

Figure 1.—Replace this figure with a new figure as shown in the accompanying Fig. 1.[3]

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[3] The new figure is not included in this report but appears in the revised method; see 1961 Book of ASTM Standards, Part 5.

Standard Method of Tension Test in Flatwise Plane of Sandwich Constructions (C 297 – 55):[2]

Title.—Change to read as follows: "Tension Test of Flat Sandwich Constructions in Flatwise Plane."

Section 1.—Add the following sentence at end of paragraph:

The test consists of subjecting a sandwich construction to tensile load normal to the plane of the sandwich, such loads being transmitted to the sandwich through thick loading blocks bonded to the sandwich facings.

Section 3.—Change the last sentence to read as follows:

The loading blocks shall be sufficiently stiff to keep the bonded facings essentially flat under load. Loading blocks not thinner than the cross-sectional dimension of the specimen have been found to perform satisfactorily.

Section 7.—Change to read as follows:

7. (*a*) Calculate the flatwise tensile strength as follows:

$$\text{Flatwise tensile strength} = \frac{\text{maximum load}}{\text{gross cross-sectional area}}$$

(*b*) For open-celled or gridded type cores, such as honeycomb where failure occurs in the bond between core and facing, the strength per unit length of adhesive fillet shall be calculated as follows:

$$\text{Strength of adhesive fillet} = \frac{\text{flatwise tensile strength}}{\text{fillet length per unit core area}}$$

Fillet length per unit core area can be found by consideration of the core cell geometry; for core with hexagon cells it has been found that fillet length per unit core area equals four divided by the cell size.

Standard Method of Test for Edgewise Compressive Strength of Flat Sandwich Constructions (C 364 – 57):[2]

Section 5.—Change the second sentence to read as follows:

This may be done (1) by fitting the specimen snugly in round steel bars slotted axially to their diameter; such bars shall have a diameter not less than the thickness of the sandwich plus ¼ in.; or (2) by using clamps made of rectangular steel bars fastened together so as to clamp the specimen lightly between them.

Section 7.—Change to read as follows:

7. Apply the load through apparatus designed to distribute the load properly to each facing and uniformly in each facing. A suitable apparatus is shown in Fig. 1. The load may be considered to be distributed properly if the strains measured on each facing are within 5 per cent of each other in the early stages of loading. It is essential that strains be measured in the early stages to avoid widely varying results due to different effective eccentricities which occur if strains are not properly balanced. Strains should be measured to the nearest 0.01 per cent by means of a strain gage having a gage length not greater than two-thirds the unsupported length of the specimen. If a complete stress-strain diagram is desired, these data may be taken throughout the test. The load shall be applied through constant rate of movement of the movable head of the testing machine (Note) such that the maximum load will occur in between 3 and 6 min.

Retain the present Note.

New Figure.—Add a new figure as shown in the accompanying Fig. 2.[3]

REAPPROVAL OF STANDARD

The committee recommends the reapproval of the Standard Definitions of Terms Relating to Structural Sandwich Constructions (**C 274 – 53**)[2] which has stood for more than six years without revision.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Method of Test for Shear Fatigue of Sandwich Core Materials (**C 394 – 57 T**).[2]

EDITORIAL CHANGES

The committee recommends editorial changes as follows in the Standard

Methods of Test for Flatwise Compressive Strength of Sandwich Cores (C 365 - 57):[2]

Section 5.—Replace the present third sentence with the following:

> The ends may be cast or dipped in a thin layer of resin (epoxy, polyester, or furan resin has been found satisfactory) and cured at room temperature or slightly elevated temperature. For cores to be tested at elevated temperatures (up to 1600 F) sodium silicate has been found satisfactory.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Mechanical Properties of Basic Materials (W. L. Emerson, chairman).—In addition to the consideration of revisions of standards as mentioned earlier in this report, the subcommittee is studying test methods for water migration, fluid flow, impact, high temperature, and dimensional stability. Method C 272 is being balloted for changes.

Subcommittee II on Mechanical Properties of Basic Sandwich Construction (E. W. Kuenzi, chairman).—Revisions were considered and accepted as mentioned earlier in this report.

Subcommittee III on Permanence, Durability, and Simulated Service (Robert Zemer, chairman).—A new tentative method for laboratory aging was completed as referred to earlier in this report. The fourth outside exposure test program known as "Delta" was developed during the year with four companies expressing an interest in participation.

This report has been submitted to letter ballot of the committee which consists of 57 members; 39 members returned their ballots, of whom 34 have voted affirmatively, and 2 negatively.

Respectfully submitted on behalf of the committee,

STEVEN YURENKA,
Chairman.

J. P. REESE,
Secretary.

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-20
ON
ACOUSTICAL MATERIALS*

Committee C-20 on Acoustical Materials held two meetings during the year: at ASTM Headquarters in Philadelphia, Pa., on Oct. 5 and 6, 1960, and at the Granada Hotel, San Antonio, Tex. on April 17 and 18, 1961.

The committee consists of 76 individuals representing 22 producers, 11 consumers, and 32 general interest members. There are 63 total votes.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Sound Absorption and Attenuation (H. J. Sabine, chairman) is continuing its study of the box method of measuring absorption, methods of achieving uniform diffusion in reverberation rooms, and methods for allowing for diffraction effects.

Subcommittee II on Flame Resistance (Wallace Waterfall, chairman) has been in liaison with Committee E-5 on Fire Tests of Materials and Construction. The purpose of meeting in San Antonio was to watch the operation of the tunnel test at Southwest Research Inst.

Subcommittee III on Maintenance (W. M. Rees, chairman) is circulating for comment a revised method of test for repaintability of acoustical materials.

Subcommittee IV on Application (R. Lindahl, chairman) has held several task force meetings during the year. Progress is being made in an attempt to write a performance specification for mechanical suspension systems for acoustical products.

Subcommittee V on Basic Physical Properties (W. A. Jack, chairman) is revising the two proposed methods of test for light reflectance, and for airflow resistance of acoustical materials.

Respectfully submitted on behalf of the committee,

R. N. HAMME,
Chairman.

RALPH HUNTLEY,
Secretary.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

REPORT OF COMMITTEE C-21
ON
CERAMIC WHITEWARES AND RELATED PRODUCTS*

Committee C-21 on Ceramic Whitewares and Related Products held two meetings during the year: in Bedford Springs, Pa., on Sept. 29, 1960, and in Toronto, Ont. on April 25, 1961.

The committee consists of 55 voting members, of whom 27 are classified as producers, 6 as consumers, and 22 as general interest members.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto:[1]

Tentative Methods of Test:

For Thermal Shock Resistance of Glazed Ceramic Tile,
For Measuring Warpage of Ceramic Tile,
For Electrical Resistance of Conductive Ceramic Tile, and
To Indicate Bond Strength of Glazed Interior Ceramic Wall Tile in Portland Cement Installations.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature (A. S. Watts, chairman) has reviewed a series of definitions that have been submitted to the committee for promulgation.

Subcommittee II, Editorial (R. F. Geller, chairman) has thoroughly reviewed all of the Committee C-21 standards and tentatives for editorial content. Minor editorial revisions are being prepared for submission to the committee.

Subcommittee III on Fundamental Properties (W. C. Mohr, chairman) is currently developing nine test methods. The method of testing for solubility of lead from glazed surfaces is being developed in concert with many interested organizations. Methods for impact test and tensile strength are being studied. The subcommittee assisted in the development of a symposium on the current status of tensile strength work in the ceramics industry, with the American Ceramic Society.

Subcommittee IV on Clays (G. W. Phelps, chairman) is continuing its work on the particle size determinations of clays using the hydrometer and centrifuge techniques.

Subcommittee V on Nuclear Applications (J. H. Handwerk, chairman) is securing interested and qualified personnel to work on the problem of establishing standards for nuclear ceramic fuels. This subcommittee believes that partial standards can now be developed for uranium dioxide and uranium dioxide-thorium dioxide fuels. These standards would not be on the raw material but rather on the finished fabricated ceramics and would be designed to correlate testing and inspecting procedures between the manufacturer and the consumer.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] The new methods appear in the 1961 Book of ASTM Standards, Part 5.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee VI on Ceramic Tile (Arno M. Illing, chairman) held a meeting on January 19, 1960, at Princeton, N. J. as well as during the two regular meetings of Committee C-21. Four proposed test methods, mentioned earlier in this report, have been submitted to the Society for publication. Eight other methods for ceramic tile are undergoing development by the subcommittee. Data from an interlaboratory comparison of specular gloss values taken with instruments of different geometries is being prepared for publication. These data indicate that the 60-deg geometry is best for determining the matness of glazed ceramic wall tile. A test method based on these data is being prepared.

Task Group C on Non-plastics (W. P. Shulhof, chairman) held a meeting on June 29, 1960, in Atlantic City, N. J. The committee reviewed its objectives toward developing standard particle size measurement of non-clay ceramic raw materials. The first effort is an interlaboratory study using electroformed sieves down to 15 μ and a centrifuge technique. The interlaboratory study covers a one-phase system of particle size below sieve No. 325 (44 μ).

Task Group G on Nuclear Grade Graphite (W. C. Riley, chairman) held meetings in Chicago, Ill., on June 13, 1960, and at La Jolla, Calif., on January 19, 1961. The Task Group has reviewed all tests available for determining the properties of nuclear grade graphite. Eight ASTM standards are in current use and, in addition, methods for outgassing spectrum, grain size, porosity, gas evolution, and oxidation rate of graphite are used in the industry. Efforts will be discussed toward developing standard methods for these last five properties. The Task Group is preparing a set of objectives for the standardizing of fuel matrix graphite.

This report has been submitted to letter ballot of the committee, which consists of 55 voting members; 50 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

N. T. MORRISON,
Chairman.

R. C. PHOENIX,
Secretary.

REPORT OF COMMITTEE C-22
ON
PORCELAIN ENAMEL*

Committee C-22 on Porcelain Enamel held two meetings during the year: on September 15, 1960, in Williamsburg, Va., and on February 3, 1961, in Cincinnati, Ohio, during ASTM Committee Week.

There were a number of resignations and acceptances of new members during the year, bringing the total membership to 56 members of whom 53 are voting members; 30 are classified as producers, 5 as consumers, and 18 as general interest members.

NEW TENTATIVE

The committee recommends for publication as tentative the Method of Test for Spalling Resistance of Porcelain Enameled Aluminum as appended hereto.[1]

REVISION OF TENTATIVE

The committee recommends revisions as appended hereto[1] of the Tentative Definitions of Terms Relating to Porcelain Enamel (**C 286 – 58 T**), and continuation of the definitions as tentative.

REVERSION OF STANDARD TO TENTATIVE WITHOUT REVISION

The committee recommends that the Standard Methods of Test for Acid Resistance of Porcelain Enamel (**C 282 – 53**)[2] be reverted to tentative without revision. Parts of the test procedure are undergoing revision, aimed largely at improving its applicability to newer types of enamel.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Methods of Test for Abrasion Resistance of Porcelain Enamels (**C 448 – 59 T**)[3] be approved for reference to letter ballot of the Society for adoption as standard without revision.

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for more than six years without revision:

Standard Method of Test for:

- Sieve Analysis of Wet Milled and Dry Milled Porcelain Enamel (**C 285 – 54**), and
- Resistance of Porcelain Enameled Utensils to Boiling Acid (**C 283 – 54**).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentative:

Tentative Method of Test for:

- Impact Resistance of Porcelain Enameled Utensils (**C 284 – 51 T**), and
- Warpage of Porcelain Enameled Flatware (**C 314 – 53 T**).

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] The new and revised tentatives appear in the 1961 Book of ASTM Standards, Part 5.

[2] 1958 Book of ASTM Standards, Part 5.

[3] 1959 Supplement to Book of ASTM Standards, Part 5.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Research (B. J. Sweo, chairman) is continuing its work on the subjects of ceramic structural adhesives, and the role of water in development of defect-producing gas during the enameling process.

Subcommittee II on Nomenclature (E. E. Howe, chairman) initiated the task of preparing a glossary to supplement the Tentative Definitions of Terms Relating to Porcelain Enamel (C 286). Some of the terms in the current definitions are to be transferred to the glossary.

Subcommittee III on Education (L. S. O'Bannon, chairman) has extended its activity to include maintenance of liaison with appropriate organizations in other countries, and has increased its membership and facilities for reaching trade and other publications.

Subcommittee IV on Materials (J. F. Matejczyk, chairman) initiated the recommendation that the Methods of Test for Sieve Analysis of Wet Milled and Dry Milled Porcelain Enamel (C 285) be reaffirmed as standard without revision. A new Joint Task Group was formed with Subcommittee XIX of Committee A-1 on Steel to select performance tests for enameling steel, to submit them to trial in interlaboratory tests, and to analyze results, with the purpose of improving existing specifications.

Subcommittee V on Finished Products (J. C. Richmond, chairman) proposed a new Tentative Method of Test for the Spalling Resistance of Porcelain Enameled Aluminum; recommended that Methods C 284 and C 314 be continued as tentative without revisions; that Method C 282 be reverted to tentative; that Method C 448 be adopted as standard without revision; and that Method C 283 be reapproved as standard. All of these recommendations are included in the report.

This report has been submitted to letter ballot of the committee, which consists of 56 voting members; 35 returned their letter ballot, of whom 32 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. N. HARRISON,
Chairman and
Acting Secretary.

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-23
ON
SORPTIVE MINERAL MATERIALS

Committee C-23 on Sorptive Mineral Materials held one meeting during the year: on July 1, 1960, in Atlantic City, N. J.

The committee consists of 21 members, of whom 13 are classified as producers, 7 as consumers, and 1 as a general interest member.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1961 Annual Meeting, Committee C-23 presented to the Society through the Administrative Committee on Standards the recommendation that the Methods for Sampling and Evaluation of Sorptive Mineral Products Used as Floor Absorbents be published as tentative. This recommendation was accepted by the Standards Committee on July 6, 1961 and the new tentative method appears in the 1961 Book of ASTM Standards, Part 10, bearing the designation C 431 - 61 T.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Definitions and Nomenclature (H. M. Gwyn, Jr., chairman) is reviewing a series of definitions following letter ballot of the committee.

Subcommittee II on Sampling and Testing (M. G. Kramer, chairman) is reviewing interlaboratory test results reported on methods for bulk density, water solubility, water breakdown, resistance to mechanical breakdown, dust content, and water absorption of sorptive mineral materials. A method for the abrasiveness of these materials is being sought. Current industrial methods for oil absorption and fire resistance will be used by the committee.

Respectfully submitted on behalf of the committee,

P. R. IZDEPSKI,
Chairman.

R. L. SHIRLEY,
Secretary.

REPORT OF COMMITTEE C-24
ON
JOINT SEALANTS*

Committee C-24 on Joint Sealants held one meeting during the year, on June 28, 1960, at Atlantic City, N. J., during the Annual Meeting of the Society. The subcommittees held meetings on the same date.

The Advisory Subcommittee has held four meetings during the year: on June 28, 1960, at Atlantic City, N. J.; on Oct. 21, 1960, at the Society Headquarters in Philadelphia, Pa.; on Nov. 15, 1960, at the Shoreham Hotel in Washington, D. C.; and on March 9, 1961, at the offices of Skidmore, Owings and Merrill in New York, N. Y.

The committee consists of 95 members, of whom 80 are voting members; 33 are classified as producers, 27 as consumers, 20 as general interest members, and 15 as consulting members.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature (Henry Lipkind, chairman).—Mr. Lipkind accepted appointment as chairman of this subcommitte in March, 1961, succeeding George Lamb, who found himself unable to continue in office. An active program is being organized.

The subcommittee has assembled several glossaries of terms pertinent to the field of joint sealants, including those definitions already established by related ASTM committees, and is in the process of soliciting from all members of Committee C-24 suggestions for terms requiring definitions. With this information at hand it expects to establish and submit for general committee approval an initial list of definitions, which will serve as the beginning of an ever-growing nomenclature relating to sealing materials and technology.

Subcommittee II on Bulk Compounds (Francis L. Frybergh, chairman) is organized in two task groups: Task Group I on Methods of Tests consisting of three subgroups; and Task Group II on Specifications.

Task Group I on Methods of Tests:

Subgroup A on Tests for Adhesion and Accelerated Aging (B. Pozniak, chairman) is proceeding with the formulation and evaluation of tests for these properties, but due to the relative complexities of the work, results are not yet available.

Subgroup B, formerly designated "Tests on Flow, Sag, and Application Life" (Roger Humke, chairman), has redefined its interests to be "Tests for Application Rate and Flow," and has conducted experimental work aimed at improving test methods for both of these properties of bulk compounds. A procedure for determining application rate by controlled extrusion versus time has appeared promising in preliminary experiments, but will require further interlaboratory testing before it can be submitted for approval as a tentative method. Experimental work is also being done to develop more accurate methods for testing flow, preparatory to subjecting these methods to interlaboratory evaluation.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

Subgroup C on Staining Tests (A. Hockman, chairman) has completed an interlaboratory evaluation of a National Bureau of Standards test for staining and on May 15, 1961, reported favorably on the results. Indications are that this test method may be superior to that specified in American Standard 116.1. Following further testing this proposed staining test procedure for rubber-base joint sealants will be submitted to the committee membership for approval as tentative.

Task Group II on Specifications (F. Frybergh, chairman) reports that since its work is necessarily dependent on the development of approved test methods, it will await action on the procedures recommended by Task Group I before undertaking the development of specifications.

Subcommittee III on Preformed Shapes (Robert W. McKinley, chairman) held a second meeting during the year, on April 11, 1961, as guests of the duPont Co., in Wilmington, Del.

Task Group A on Architectural Seal (C. B. Monk, Jr., chairman) has had as its primary concern a comprehensive study aimed at defining the basic criteria for joint seals in architectural work. This resulted in the presentation of a paper, "Study of Architectural Joint Problems" by C. B. Monk, which will admirably serve as a guide in the preparation of specific standards. To supplement this, Mr. Monk has also prepared an "Outline of End Use Requirements of Preformed Gaskets and Tapes as Joint Sealants," and Mr. Jackson has submitted for the group's consideration a "Proposed Guide for Specifications and Joint Sealants."

Work is under way on a recommended performance specification for structural gasketing, and the group has also under consideration a specification for neoprene sponge, and for vinyl weatherstrip.

Task Group B on Underground Pipe Seals (F. Valenziano, chairman) has compiled a list of standards relating to underground pipe joints, seals, and test methods to provide a background for the development of further standards.

This report has been submitted to letter ballot of the committee, which consists of 95 members; 61 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

WAYNE F. KOPPES,
Chairman.

JULIAN R. PANEK,
Secretary.

REPORT OF COMMITTEE C-25
ON
CERAMICS FOR ELECTRONICS

Committee C-25 on Ceramics for Electronics was organized on April 28, 1960, during the annual meeting of the American Ceramic Society in Philadelphia, Pa. Two subsequent meetings were held: on Sept. 22, 1960, in Milwaukee, Wis., and on April 25, 1961, in Toronto, Ont.

The committee consists currently of 169 members, of whom 142 are voting members and 27 are consulting members. The officers are:

Chairman, Ralph L. Cook.
Vice-Chairman, Herman H. Frahme.
Secretary, Wm. W. Coffeen.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Classification and Nomenclature (A. Pincus, chairman) is working closely with the same activity of the Electronics Division of the American Ceramic Society to develop a system of classification for electronic ceramics having a series of definitions of terms relating to this field.

Subcommittee III on General Methods of Test (J. L. Pentecost, chairman) is soliciting the electronics industry to determine those ceramic test methods that need standardization.

Subcommittee IV on Electrical Insulating Materials (H. D. Taylor, chairman) has initiated an interlaboratory test program to develop standard methods for compressive strength and flexural strength of ceramic insulators. Consideration is being given to tests for the measurement of dielectric constant and loss at microwave frequencies. Liaison is being maintained with ASTM Committees C-21 on Whitewares, D-9 on Electrical Insulating Materials, and F-1 on Materials for Electron Tubes and Semiconductor Devices.

Subcommittee V on Ferrolectrics (Roland R. Roup, chairman) has organized task groups on electrical test methods, process controls, and raw materials. A survey of electrical test methods used in the ceramics field has been initiated.

Subcommittee VI on Non-Metallic Magnetic Materials (D. L. Fresh, chairman):

Task Group A on Computer Applications (J. R. Brown, Jr., chairman) held a meeting on May 24, 1961, in Washington, D. C. in order to further work on a method of testing coincident current memory cores.

Task Group B on Microwave Applications (E. T. Wierman, chairman) met on May 12, 1960, in Boulder, Colo. Three proposed methods are nearing completion for the complex dielectric constant of non-metallic magnetic materials at microwave frequencies, ferromagnetic resonance linewidth, and gyromagnetic ratio of non-metallic magnetic materials.

Task Group C on Pulse Transformer Applications (C. W. Lundberg, chairman) is being organized.

Subcommittee VII on Semiconductor Materials (W. E. Hauth, Jr., chairman) is initiating a program to develop tests for fundamental data on ceramic semiconductors comparable to that currently available for single crystal materials.

Work is also under way on materials for both ohmic and non-ohmic resistor applications.

Subcommittee VIII on Composites (Morris Berg, chairman) is organizing six task groups to cover films and coatings, laminates, fiber composites, impregnates, bonded particulates, and semiconductors.

Subcommittee IX on Research (Gilbert Goodman, chairman) is reviewing the scope of research in the electronic ceramics field for possible new areas of work that may be of value to the work of the committee.

Respectfully submitted on behalf of the committee,

R. L. COOK,
Chairman.

W. W. COFFEEN,
Secretary.

REPORT OF COMMITTEE D-1

ON

PAINT, VARNISH, LACQUER, AND RELATED PRODUCTS*

Committee D-1 on Paint, Varnish, Lacquer, and Related Products held two meetings during the year: on June 27 to 29, 1960, in Atlantic City, N. J., in connection with the Annual Meeting of the Society, and in Roanoke, Va., on Feb. 13 to 15, 1961.

At the June, 1960, meeting, Walter C. Granville, Industrial Color Consultant, presented a very interesting paper on "The Relation Between Color Systems and Color Usage." It covered such matters as popular trends in color as related to paints, roofing materials, and other similar items. The author pointed out that colors should be chosen in terms of a particular situation or environment rather than from the relationship of the colors to each other. Background effects on colors should also be given consideration in the development of a color scheme.

At the February, 1961, meeting James McCallum of the Beckman Instruments Co., presented a talk on "Gas Chromatography in the Analysis of Paints," pointing out the basic principles of gas chromatography and techniques for its use in analytical work with paint materials.

Committee D-1 has continued its close cooperation with the Federation of Societies for Paint Technology and as a result 112 standards have been jointly approved by both organizations. The joint Federation-ASTM Committee on Paint, Varnish, and Lacquer (B. G. Brand, chairman), serves as the permanent liaison between the two organizations.

The Committee on Intercommittee Relations (J. C. Weaver, chairman) is composed of the official representatives of Committee D-1 on other ASTM technical committees. The committee thus serves to maintain close contact between Committee D-1 and those committees whose activities are of interest to Committee D-1. In the past year, a representative of Committee D-1 has been appointed to the new Committee E-18 on Sensory Evaluation of Materials and Products.

At the February, 1961, meeting, Henry A. Gardner and Robert I. Wray were unanimously elected Honorary Members of Committee D-1.

NEW STANDARDS

The methods listed below currently appear in the Standard Methods of Testing Drying Oils (D 555 - 58),[1] or the Tentative Methods of Testing Fatty Acids Used in Protective Coatings (D 1467 - 59 T),[2] or in both as indicated. The committee recommends that these methods be published as separate standards with the exception of the test for hydroxyl value which is recommended for publication as tentative. The sections of Methods D 555 and D 1467 where these tests currently appear are indicated below; the methods themselves are not appended to this report. In several cases it has been necessary to make minor changes in the methods as indicated in order to complete them for publication as separate standards.[3]

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] 1958 Book of ASTM Standards, Part 8.

[2] 1959 Supplement to Book of ASTM Standards, Part 8.

Standard Method of Test for Specific Gravity of Drying Oils, Varnishes, Resins, and Related Materials at 25/25 C (Present Sections 4 to 9 of Methods D 555):

New Section.—Add the following new sections on scope and precision:

Scope.—This method is applicable to drying oils, varnishes, alkyd resins, fatty acids and related materials. The procedures described here are recommended where precision is required in the determination of specific gravity, such as establishing specifications or referee work. Procedures are included for the following:

(*1*) Materials with a viscosity of 40 stokes or less use the Leach type pycnometer.

(*2*) Materials with a viscosity in excess of 40 stokes use the Hubbard type pycnometer.

(*3*) Materials that are solid or semi-solid at 25 C but liquid at slightly higher temperatures use the Hubbard type pycnometer.

The weight-per-gallon cup method is satisfactory for most routine laboratory work although the precision does not equal that of the pycnometer. The procedure described here for the use of the Hubbard type pycnometer is applicable to the weight-per-gallon cup or the Method of Test for Density of Paint, Varnish, and Related Products (ASTM Designation: D 1475) may be used.

Hydrometers provide a rapid method for checking specific gravity and are generally satisfactory for routine control work. However, the precision of the hydrometer method is not adequate where accurate results are required. The Method of Test for Specific Gravity of Petroleum Products (ASTM Designation: D 1298) describes the procedure for use of the hydrometer in determination of specific gravity.

NOTE.—Hydrometers are normally calibrated to read specific gravity at 15.5/15.6 C. The ASTM specifications for specific gravity of drying oils and fatty acids are given at 25/25 C. Difficulties have been experienced in obtaining hydrometers calibrated at 25/25 C.

Precision.—(*a*) Fourteen laboratories participating in the AOCS Smalley check series reported single results on 6 oils for a total of 84 results and a calculated standard deviation of 0.00049.

(*b*) Reproducibility, 95 per cent confidence limits: Single determinations made in two different laboratories shall not differ by more than 0.0013.

Standard Method of Test for Saponification Value of Drying Oils and Fatty Acids (Present Sections 14 to 17 of Methods D 555 and Sections 19 to 23 of Methods D 1467):

New Section.—Add the following new section on scope:

Scope.—This method covers the determination of the saponification value of drying oils, bodied oils, and fatty acids.

Standard Method of Test for Unsaponifiable Matter in Drying Oils and Fatty Acids (Present Sections 18 to 21 of Methods D 555 and Sections 24 to 28 of Methods D 1467):

New Section.—Add the following new section on scope:

Scope.—This method covers a procedure for determining the unsaponifiable matter present in all natural and synthetic drying oils and their fatty acids and any other fatty acids used in protective coatings.

Standard Method of Test for Iodine Value of Drying Oils and Fatty Acids (Present Sections 22 to 25 of Methods D 555 and 14 to 18 of Methods D 1467):

New Section.—Add the following new section on scope and a new Note 2 under Definition:

Scope.—This method describes the Wijs procedure for determination of unsaturation (iodine value) in drying oils. It is applicable to all natural and synthetic drying oils and their fatty acids.

NOTE 2.—The Wijs iodine value method is not reliable for tall oil fatty acids containing appreciable quantity of rosin acids.

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[3] The new standards appear in their entirety in the 1961 Book of ASTM Standards, Part 8.

Standard Method of Test for Loss on Heating of Drying Oils (Present Sections 26 and 27 of Methods D 555):

New Section.—Add the following new section on scope:

Scope.—This method determines moisture and any other material that is volatile under the conditions of the test. It is applicable to all natural drying oils (Notes 1 and 2).

NOTE 1.—Care must be taken to keep the surface of the oil blanketed with inert gas throughout the test to prevent oxidation of the oil.

NOTE 2.—For solutions of drying oils in volatile organic solvents it is recommended that the Methods of Test for Nonvolatile Content of Resin Solutions (ASTM Designation: D 1259) or for Non-volatile Content of Varnishes (ASTM Designation: D 1644) be used.

Standard Method of Test for Foots in Raw Linseed Oil (Present Sections 31 to 37 of Methods D 555):

Revised Sections.—Revise the sections on scope and definition to read as follows:

Scope.—This method is designed to cover the determination of foots in raw linseed oil.

Definition.—(*a*) *Foots* is a term that originally was used to describe those solid impurities that precipitate from raw linseed oil during storage and which then settle to the bottom or "foot" of a storage tank.

(*b*) In this test the term foots denotes material insoluble in a mixture of equal parts of acetone and the oil under test, and insoluble in calcium chloride solution, under the specific conditions of the test.

New Sections.—Add the following two new sections on Report and Precision:

Report.—Report the results to the first decimal place.

Precision.—Adequate precision data are not available. Some oils yield relatively good precision data; other oils, at comparable foots levels, yield relatively poor data. The production methods used are the primary causes of this variation.

Standard Method of Test for Quantitative Determination of Break in Drying Oils (Present Sections 38 to 42 of Methods D 555):

New Section.—Add the following new section on scope:

Scope.—This method is applicable to nonsubstituted raw and refined drying or fatty oils that have not been substantially polymerized, oxidized or chemically modified.

Definition.—Revise the definition of break to read as follows:

Break in drying oils is that material which may be rendered insoluble when the oil is heated to polymerization or varnish-making temperatures.

Standard Method of Test for Acetone Tolerance of Heat-Bodied Drying Oils (Present Sections 43 to 47 of Methods D 555).

Standard Method of Test for Gel Time of Drying Oils (Present Sections 48 to 51 of Methods D 555):

New Section.—Add the following new section on precision:

Precision.—At 67 per cent confidence limits, standard deviation within a laboratory is 5 sec and between laboratories 25 sec.

Standard Method of Test for Tung Oil Quality (Present Sections 52 to 55 of Methods D 555).

Standard Method of Test for Drying Properties of Drying Oils (Present Sections 56 to 59 of Methods D 555):

New Sections.—Add the following new sections on scope, definitions, and precision:

Scope.—This method determines the drying characteristics of drying oils.

Definitions: (*a*) *Set-to-Touch Time.*—The time required, under the conditions outlined, for the oil to reach a point where the adhesion to an external object is less than the internal cohesion of the film.

(*b*) *Dry Time.*—The time required, under the conditions outlined, for the oil film to reach the end point described herein.

Precision: (*a*) *Set-to-Touch Time.*—Collaborative tests indicate that repeated measurements in the same laboratory on set-to-touch time should agree within 0.2 hr. Different laboratories should agree within 0.4 hr.

(*b*) *Time of Drying.*—Because of the subjective nature of the time of drying test, the agreement to be expected between laboratories depends upon their understanding of the terms used, and cannot be established with certainty. Within a laboratory, the agreement depends upon the oil being tested, some being much sharper in their end point than others, but duplicate determinations should agree within 10 per cent of the time of drying.

Standard Method of Test for Ash in Drying Oils and Fatty Acids (Present Sections 60 to 62 of Methods D 555 and Sections 40 to 43 of Methods D 1467):

Standard Method of Test for Matter Insoluble in Chloroform in Oiticica Oil (Present Sections 64 to 66 of Methods D 555):

New Section.—Add the following new section on scope:

Scope.—This method of test covers the matter insoluble in chloroform of oiticica oil. It may also be used for other drying oils.

Standard Method of Test for Heat Bodying Rate of Drying Oils (Present Sections 77 to 81 of Methods D 555).

Standard Method of Test for Maleic Diene Value of Drying Oils (Present Sections 82 to 86 of Methods D 555).

Standard Method of Test for Titer of Fatty Acids (Present Sections 7 to 9 of Methods D 1467).

Standard Method of Test for Acid Value of Fatty Acids (Present Sections 10 to 13 of D 1467):

New Sections.—Add the following new sections on scope and precision:

Scope.—This method measures the acidity or amount of free fatty acids and is applicable to all fatty acids.

Precision.—In a collaborative study involving four laboratories with two operators in each laboratory and three samples of fatty acids the following precision, at 95 per cent confidence limits, was obtained:

(*a*) *Repeatability.*—Two single determinations performed in one laboratory shall not differ by more than 2.3.

(*b*) *Reproducibility.*—Single determinations performed in two different laboratories shall not differ by more than 2.4.

Standard Method of Test for Color After Heating of Fatty Acids (Present Sections 29 to 32 of Methods D 1467):

New Section.—Add the following new section on significance of test:

Significance of Test.—The color of a fatty acid is readily affected by heat and oxidization. Variations on degree of heat, time of heat and exposure to atmosphere during heating have a marked effect on the color obtained, therefore, conformity to the equipment and procedure outlined in the method is essential to accuracy and precision.

NEW TENTATIVES

The committee recommends for publication as tentative the following specifications and methods as appended hereto:[4]

Tentative Specifications for:

- Distilled Coconut Fatty Acids,
- Distilled Corn Fatty Acids,
- Fractionated and Distilled Cottonseed Fatty Acids, and
- Asbestos-Cement Shingle Blanks to be Used as Panels in Weathering Tests of Latex and Emulsion Paints.

Tentative Methods of:

- Chemical Analysis of Strontium Chromate Pigment,
- Test for Water Soluble Matter in Iron Oxide Pigment by Specific Resistance Method,
- Chemical Analysis of Basic Lead Silico Chromate,
- Test for Total Chlorine Content of Epoxy Resins,
- Reporting Paint Film Failures Characteristic of Exterior Latex Paints, and
- Test for Package Stability of Latex Paint.

Tentative Method of Test for Hydroxyl Value of Fatty Oils and Acids (Present Sections 67 to 70 of Methods D 555):

Definition.—Omit the present definition in Section 67.

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[4] The new tentatives appear in the 1961 Book of ASTM Standards, Part 8.

New Sections.—Add the following new sections on scope and precision.

Scope.—(*a*) This method covers the determination of the hydroxyl content of castor oil, dehydrated castor oil, and their derivatives.

(*b*) This method may also be used for other fatty products such as; fatty alcohols, mono and diglycerides and hydroxystearic acid but the precision will not necessarily be as indicated.

Precision.—The following data should be used for judging the acceptability of the results on hydroxyl value (95 per cent confidence level):

(*a*) *Repeatability.*—Duplicate results by the same operator should not be considered suspect unless they differ by more than 2.4.

(*b*) *Reproducibility.*—The result submitted by each of two laboratories should not be considered suspect unless two results differ by more than 3.0.

REVISION OF TENTATIVE

The committee recommends revision as follows of the Tentative Method of Test for Acidity in Lacquer Solvents and Diluents (**D 1613 - 58 T**),[1] and continuation of the method as tentative:

Note 1.—Change to read as follows:

NOTE 1.—Isopropyl alcohol or 190-proof ethyl alcohol conforming to formula No. 3A of the U. S. Bureau of Internal Revenue is suitable for use as the solvent. The use of methyl alcohol is not recommended.

TENTATIVE REVISION OF STANDARD

The committee recommends the following two definitions as a tentative revision of the Standard Definitions of Terms Relating to Paint, Varnish, Lacquer, and Related Products (**D 16 - 59**):[2]

Durability.—A relative term indicating degree of permanency. It may be applied to individual protective, decorative, or functional properties, for example, "the durability of gloss," but if used in a general way, for example, "the excellent durability of a paint," implies the ability of the described coating to retain, to the indicated degree, all of the properties required for the continued service of the coating.

Lacquer.—A coating composition which is based on synthetic thermoplastic film-forming material dissolved in organic solvent and which dries primarily by solvent evaporation. Typical lacquers include those based on nitrocellulose, other cellulose derivatives, vinyl resins, acrylic resins, etc.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for:

Raw Tung Oil (**D 12 - 59 T**),[2]
Boiled Linseed Oil (**D 260 - 58 T**),[1] and
Basic Lead Silico-Chromate (**D 1648 - 59 T**).[2]

Tentative Methods of Test for:

Viscosity Reduction Power of Hydrocarbon Solvents (**D 1311 - 54 T**),[1]
No-Smear Time of Traffic Paint (**D 1359 - 55 T**),[1]
Sampling Liquid Oils and Fatty Acids Commonly Used in Paints, Varnishes, and Related Materials (**D 1466 - 57 T**),[1]
Effect of Staining Agents on Organic Finishes Used in the Transportation Industry (**D 1540 - 58 T**),[1]
Phthalic Acid Isomers and Benzoic Acid in Alkyd Resins and Esters (**D 1651 - 59 T**),[2] and
Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments (**D 1654 - 59 T**).[2]

ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that the following tentative be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

Tentative Method of Test for Kauri-Butanol Value of Solvents (D 1133 - 54 T):[1]

Section 8.—Change to read as follows:

8. *Precision.*—Duplicate results in the range of 30 to 90 should not be considered suspect

unless they differ by more than the following amounts (95 per cent probability):

Repeatability	Reproducibility
$0.01K - 0.1$	$0.03K + 1.0$

where:

K = average kauri-butanol value.

Methods of Testing Fatty Acids Used in Protective Coatings (D 1467 - 59 T):[2]

Revise as appended hereto.[5] This revision is contingent upon the establishment as standards of the several methods referred to as indicated earlier in this report under New Standards.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Method of Test for Flash Point by Tag Closed Tester (D 56 - 56) (Jointly with Committee D-2):[1]

Section 1.—Change to read as follows:

1. This method of test is intended for determining the flash point of all mobile liquids flashing below 175 F (79 C), except products classed as fuel oils.

Appendix II, Section A6.—In the requirements for purity insert after the words "95 per cent min," the following "(11.23 C freezing point, min)."

Methods of Sampling and Testing Lacquer Solvents and Diluents (D 268 - 59):[2]

Sections 20 to 24.—Delete those sections which describe the toluene (toluol) dilution ratio test and replace by a reference to the Tentative Method of Test for Dilution Ratio in Cellulose Nitrate Solutions for Active Solvents, Hydrocarbon Diluents, and Cellulose Nitrates (D 1720 - 60 T).

Standard Specifications for Leaded Oxide (D 80 - 41):[1]

Section 2.—Change the table of requirements for composition and properties of dry pigment to read as follows:

	35 per cent Leaded
Zinc oxide, per cent	61 to 67
Matter soluble in water, max, per cent	1.0
Moisture and other volatile matter, max, per cent	0.5
Total zinc oxide and basic lead sulfate, min, per cent	98
Basic lead sulfate (containing 15 to 28 per cent PbO):	
Min, per cent	33
Max, per cent	37
Coarse particles (total residue retained on a No. 325 (44-micron) sieve, max, per cent	1.0

Standard Specifications for Petroleum Spirits (Mineral Spirits) (D 235 - 59):[2]

Section 2.—Change the distillation requirements to read as follows:

Percentage recovered at:	
350 F (177 C), using Method D 86, min	50
357 F (180.5 C), using Method D 1078, min	50
End Point:	
Using Method D 86, max	410 F (210 C)
Using Method D 1078, max	(417 F) 214 C

Section 3(d).—Change to read as follows:

(*d*) *Distillation.*—Method of Test for Distillation of Petroleum Products (ASTM Designation: D 86) or Method of Test for Distillation Range of Lacquer Solvents and Diluents (ASTM Designation: D 1078). Distillation data obtained by Method D 86 can be converted to Method D 1078, or vice versa, by employing the following equations:[5a]

$$\text{D 1078, in deg Cent} = 1.025\,(\text{D 86, in deg Cent}) - 0.993$$

$$\text{D 86, in deg Cent} = 0.975\,(\text{D 1078, in deg Cent}) + 0.969$$

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[5] The revised standards appear in the 1961 Book of ASTM Standards, Part 8.

[5a] These equations are based on data obtained in an interlaboratory study of Methods D 86.

and D 1078 as reported in an article entitled, "Cooperative Interlaboratory Study of ASTM Distillation Methods D 86 and D 1078," by T. R. Donlan, which is now pending publication.

Standard Methods of Test for Nonvolatile Content of Resin Solutions (D 1259 - 58):[1]

Revise as appended hereto.[5] The results of the cooperative tests in the interlaboratory study of these revised methods are shown in Tables I and II.

Standard Specifications for Industrial 90 Benzene for Use in Paint, Varnish, Lacquer, and Related Products (D 361 - 36): Revise as appended hereto.[5]

Standard Specifications for Industrial Grade Toluene for Use in Paint, Varnish, Lacquer, and Related Products (D 362 - 36): Revise as appended hereto.[5]

Standard Specifications for Industrial Grade Xylene or Solvent Naphtha for Use in Paint, Varnish, Lacquer, and Related Products (D 364 - 36): Revise as appended hereto.[5]

Standard Methods of Testing Drying Oils (D 555 - 58):

Revise as appended hereto.[5] This revision is contingent upon the establishment as standards of the several methods referred to as indicated earlier in this report under New Standards.

Standard Method of Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products (D 609 - 52): Revise as appended hereto.[5]

Standard Method of Test for Acid Value of Varnishes (D 1639 - 59):[2] Revise as appended hereto.[5]

WITHDRAWAL OF STANDARDS

The committee recommends the withdrawal of the following standards for the reasons indicated:

Standard Method of Test for Relative Dry Hiding Power of White Pigments in a Linseed Oil Vehicle (D 406 - 39).[1] This method has been superseded by Method D 1738 - 60 T.

Standard Method of Test for Nitrocellulose Diluting Power of Hydrocarbon Solvents (D 1134 - 53).[1] This method is now included in Method D 1720 - 60 T.

WITHDRAWAL OF TENTATIVE

The committee recommends the withdrawal of the Tentative Specifications for Heavy Petroleum Spirits (Heavy Mineral Spirits) (**D 965 - 59 T**)[2] because the specifications are obsolete.

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Standard Specifications for:

Wood to be Used as Panels in Weathering Tests of Paints and Varnishes (**D 358 - 55**),
Oiticica Oil (Permanently Liquid) (**D 601 - 55**),
Raw Castor Oil (**D 960 - 52**), and
High-Gravity Glycerin (**D 1257 - 55**).

Standard Methods of Test for:

Specific Gravity of Pigments (**D 153 - 54**),
Oil Absorption of Pigments by Spatula Rub-out (**D 281 - 31**),
Consistency of Exterior House Paints and Enamel-Type Paints (**D 562 - 55**),
Evaluating Degree of Resistance to Rusting Obtained with Paint on Iron or Steel Surfaces (**D 610 - 43**),
No-Pick-up Time of Traffic Paint (**D 711 - 55**),
Conducting Road Service Tests on Traffic Paint (**D 713 - 46**),
Producing Films of Uniform Thickness of Paint, Varnish, Lacquer, and Related Products on Test Panels (**D 823 - 53**),
Water Immersion Test of Organic Coatings on Steel (**D 870 - 54**),
Laboratory Test for Degree of Resistance of Traffic Paint to Bleeding (**D 969 - 54**),
Heptane Number of Hydrocarbon Solvents (**D 1132 - 53**),

Roundness of Glass Spheres (**D 1155 – 53**),

Measurement of Dry Film Thickness of Non-Magnetic Coatings of Paint, Varnish, Lacquer, and Related Products Applied on a Magnetic Base (**D 1186 – 53**),

Measurement of Wet Film Thickness of Paint, Varnish, Lacquer, and Related Products (**D 1212 – 54**),

Crushing Resistance of Glass Spheres (**D 1213 – 54**),

TABLE I.—RESULTS OF COOPERATIVE TESTS BY FOIL METHOD FOR NONVOLATILE CONTENT OF EPOXY RESIN SOLUTIONS (METHOD B, 2 HR IN FORCED-VENTILATION OVEN AT 105 C).

Laboratory	Jones-Dabney Hard Epoxy Resin Solution, 60 per cent Solids		Jones-Dabney Epoxy Resin Solution, 50 per cent Solids		Shell Epon, 836-C-75 Resin Solution, 75 per cent Solids		Shell Epon, 1007-CT-55 Resin Solution, 52 per cent Solids		Range Within Laboratory, Average, per cent
	Average, per cent	Range, per cent	Average, per cent	Range, per cent	Average, per cent	Range, per cent	Average, per cent	Range, per cent	
Aberdeen Proving Ground	59.2	0.0	...	...	...	...	51.6	0.1	0.1
Allied Chemical Corp.	59.5	0.1	49.6	0.1	75.5	0.2	51.9	0.5	0.2
Hercules Powder Co.	59.5	0.1	49.7	0.3	75.5	0.1	52.1	0.7	0.3
Shell Chemical Corp.	59.3	0.2	49.6	0.1	75.5	0.2	52.3	0.2	0.2
Sherwin-Williams Co.	59.6	0.1	49.9	0.4	75.6	0.1	52.4	0.0	0.2
Average of Average Results	59.4	...	49.7	...	75.5	...	52.1	...	...
Range of Average Results	0.4	...	0.3	...	0.1	...	0.8	...	...
Average Range	...	0.1	...	0.2	...	0.2	...	0.3	...

TABLE II.—COMPARISON OF GRAVITY-CONVECTION AND FORCED-VENTILATION OVENS FOR DETERMINING NONVOLATILE CONTENT OF NON-HEAT-REACTIVE RESIN SOLUTIONS (METHOD A, 30 MIN AT 105 C).

Laboratory	Baking-Type Tall Oil Alkyd in Mixed Xylol and VM & P Naphtha, 50 per cent Solids		Air-Dry Linseed Alkyd in Mineral Spirits, 50 per cent Solids		Air-Dry Soya Alkyd in Mineral Spirits, 50 per cent Solids	
	Type Oven		Type Oven		Type Oven	
	Forced Ventilation, Average per cent	Gravity Convection, Average per cent	Forced Ventilation, Average per cent	Gravity Convection, Average per cent	Forced Ventilation, Average per cent	Gravity Convection, Average per cent
Aberdeen Proving Grounds	49.6	49.7	50.0	50.0	51.3	51.4
Allied Chemical Corp.	49.7	49.7	49.8	49.6	51.2	51.1
American Cyanamid	49.9	50.1	50.1	50.2	51.3	51.4
Average of Average Results	49.7	49.8	50.0	49.9	51.3	51.3
Range of Average Results	0.3	0.4	0.3	0.6	0.1	0.3
Difference in Average Results by the Two Methods	...	+0.1	...	−0.1	...	0.0

Solvent Tolerance of Amine Resins (**D 1198–55**),

Color of Clear Liquids (Platinum-Cobalt Scale) (**D 1209 – 54**),

Fineness of Dispersion of Pigment-Vehicle Systems (**D 1210 – 54**),

Sampling and Testing High-Gravity Glycerin (**D 1258 – 55**),

Residual Odor of Lacquer Solvents and Diluents (**D 1296 – 55**), and

Single and Multi-Panel Forms for Recording Results of Exposure Tests of Paints (**D 1150 – 55**).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Methods of Test for:

Knoop Indentation Hardness of Paint, Varnish, and Lacquer Coatings (**D 1474 – 57 T**),

Epoxy Content of Epoxy Resins (**D 1652 – 59 T**), and

Moisture Vapor Permeability of Organic Coating Films (**D 1653 – 59 T**).

CONTINUATION OF TENTATIVE REVISION

The committee recommends that the tentative revision covering a method of test for heat-bleach of drying oils of the Standard Methods of Testing Drying Oils (D 555 – 58)[1] be continued without revision.

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following standards and tentatives:

Standard Specifications for Amyl Acetate (Synthetic) (85 to 88 per cent Grade) (D 318 – 58):[1]

Section 3(d).—Change lines 5 and 6 to read: "Distillation Thermometer 102 C having a range of 123 to 177 C."

Standard Specifications for Ethylene Glycol Monobutyl Ether (D 330 – 56):[1]

Section 3(d).—Change lines 5 and 6 to read: "Distillation Thermometer 103 C having a range of 148 to 202 C."

Standard Specifications for Ethylene Glycol Monoethyl Ether (D 331 – 56):[1]

Section 3(d).—Change lines 5 and 6 to read: "Distillation Thermometer 102 C having a range of 123 to 177 C."

Standard Specifications for Acetate Ester of Ethylene Glycol Monoethyl Ether (95 per cent Grade) (D 343 – 56):[1]

Section 3(d).—Change lines 5 and 6 to read: "Distillation Thermometer 102 C having a range of 123 to 177 C."

Standard Method of Evaluating Degree of Resistance to Chalking of Exterior Paints of the Linseed-Oil Type (D 659 – 44):[1]

Title.—Delete "of the Linseed-Oil Type."

Section 1.—Delete "linseed-oil type" in line 4, and "on wood surfaces" in line 5.

Section 2.—Add to the end of the last sentence: "or other means."

Section 4.—Add to the end of the paragraph: "There are also mechanical devices which may be used to accomplish the results."

New Footnote.—Add a new footnote referenced to the new sentence in Section 4 to read: "An example is the Erickson chalk rate tester."

Section 5(d).—Delete the last two sentences.

Standard Method of Evaluating Degree of Resistance to Checking of Exterior Paints of the Linseed-Oil Type (D 660 – 44):[1]

Title.—Delete "of the Linseed-Oil Type."

Section 1.—Delete "linseed-oil type" in line 4, and "on wood surfaces" in line 5.

Section 4(d).—Delete "Linseed-oil" and change "often" to "may" in line 1.

Standard Method of Evaluating Degree of Resistance to Cracking of Exterior Paints of the Linseed-Oil Type (D 661 – 44):[1]

Title.—Delete "of the Linseed-Oil Type."

Section 1.—Delete "linseed-oil type" in line 4, and "on wood surfaces" in line 5.

Section 4(d).—Delete "Linseed-oil" and change "often" to "may" in line 1.

Section 4(e).—Insert "wood panels" after "examining" in line 1.

Standard Method of Evaluating Degree of Resistance to Erosion of Exterior Paints of the Linseed-Oil Type (D 662 – 44):[1]

Title.—Delete "of the Linseed-Oil Type."

Section 1.—Delete "linseed-oil type" in line 4, and "on wood surfaces" in line 5.

Section 2.—Add the words "or undercoat" after the word "substrate" in lines 3 and 5.

Standard Methods of Evaluating Degree of Resistance to Flaking (Scaling) of Exterior Paints of the Linseed-Oil Type (D 772 – 47):[1]

Title.—Delete "of the Linseed-Oil Type."

Section 1.—Delete "linseed-oil type" in line 4, and "on wood surfaces" in line 5.

Section 2.—Add the words "or blistering" after the word "checking" in line 7.

Standard Method of Test for Distillation Range of Lacquer Solvents and Diluents (D 1078 – 58):[1]

Section 4(d).—Delete the words "boiling below 115 C" in line 7.

Table I.—Add Thermometers 102C through 107C.

Tentative Recommended Practice for Visual Evaluation of Color Differences of Opaque Materials (D 1729 – 60 T):[6]

Title.—Change to read: "Tentative Method for Visual Evaluation of Color Differences of Opaque Materials."

Section 1.—Delete "recommended practice" and insert the word "method" in line 1.

Section 2(a).—Delete "recommended practice" and insert the word "method" in lines 2 and 3.

Section 3(e)(1).—Delete the second sentence and add: "Units to be used in this method must meet the spectral quality limits set forth in this method."[4]

Footnote 4.—Change to read as follows:

> The following units are known to meet the spectral quality limits set forth in this method: (AAA) Macbeth North Daylight and (AA) Macbeth DeLuxe Examolite. These units are obtainable from the Macbeth Daylighting Co., P. O. Box 950, Newburgh, N. Y."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[7]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Drying Oils (D. S. Bolley, chairman) prepared the tentative specifications for corn, coconut, and cottonseed fatty acids, the revision of Methods D 555 and D 1467, and the 18 methods of testing drying oils and fatty acids. The subcommittee is in the process of preparing a replacement for the Specifications for Raw Castor Oil (D 960).

Subcommittee IV on Traffic Paint (W. W. Burr, chairman) is conducting a program for obtaining state and municipal traffic paint purchasing specifications which should indicate which test methods are most often used and therefore most important. This study should also indicate which methods of Subcommittee IV need revision and point to the need for new tests. The subcommittee is making a preliminary study on an infrared method for comparing traffic paint shipments with approved samples. A method for measuring reflectance of wet traffic paint is under consideration.

Subcommittee V on Solvents (E. F. Rogers, chairman) has established a study group to evaluate improved methods for defining solvent power of hydrocarbons. The group on oxygenated solvents has developed and submitted to Subcommittee X for comments a method for measuring the optical properties of the 500 platinum-cobalt color standard. Task groups have been established for statistically evaluating the precision of Methods D 1364, D 1613, and D 1617.

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[6] 1960 Supplement to Book of ASTM Standards, Part 8.

[7] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Active work is continuing on the development of specifications and test methods for vinyl acetate, acetaldehyde, formaldehyde, and pentaerythritol. Also, a standing task force was established to follow and advise ASTM on Federal solvent specifications and test methods. The subcommittee prepared the revised statement of precision for Method D 1133, revised temperature specifications for Specification D 235, and the revised Specifications D 361, D 362, and D 364.

Subcommittee VI on Definitions (F. B. Steig, Jr., chairman) prepared the definition on "durability" and has accepted with slight editorial change a definition for "lacquer" proposed by Subcommittee XXV. The subcommittee has under consideration proposed definitions for "epoxy," "epoxy group," "epoxy resin," "urethane coating," and "fire-retardant paint." It is also reviewing a number of definitions of Committee D-16, on Industrial Aromatic Hydrocarbons, which have been suggested for revision.

Subcommittee VII on Accelerated Tests for Protective Coatings (N. B. Garlock, chairman) is cooperating with Subcommittee XXV on a new method of test for perspiration resistance of organic coatings and prepared the revision of Method D 609. Work is in progress on types of standards for very fine blisters, methods of testing under conditions of 100 per cent relative humidity, and continuous condensation on test panels, accelerated testing of house paints for moisture-blister resistance, a test for cracking due to plasticizer migration, thermal shock test for cracking of finishes, and the collection of photographs to illustrate different degrees of types of failure after weathering of paints. It is proposed to limit the search at the present time to checking. The subcommittee also recommends that Tentative Methods D 1540 and D 1654 be adopted as standard.

Subcommittee VIII on Methods of Chemical Analysis of Paint Materials (W. H. Madson, chairman) prepared the Tentative Methods for Chemical Analysis of Strontium Chromate Pigment, and for Basic Lead Silico Chromate, and the Methods for Water-Soluble Matter in Iron Oxide Pigments by Specific Resistance Method. An attempt is being made to consolidate the existing twenty methods for determining non-volatile content of solvents. Test results to date indicate that none of the present methods is suitable for determining low concentrations of lead in paint. The subcommittee is exploring the possibility of developing a method for the analysis of a whole paint. Also, work is under way for developing methods for determining water-soluble matter in a number of white pigments by the specific resistance method.

Subcommittee IX on Varnish (B. G. Brand, chairman) is preparing revisions of Methods D 1639, D 1544, and D 1469. The subcommittee is continuing its work on a proposed tentative method of test for clarity and cleanness of paint liquids. The subcommittee has suggested the possibility of consolidating the existing 37 methods or specifications for drying times or drying properties. A similar proposal has been made in regard to the 19 methods for specific gravity. A review of print-test methods has indicated the desirability of coordinating Federal Standard 141 Method 6211 and ASTM Method D 1645.

Subcommittee X on Optical Properties (H. K. Hammond, III, chairman) prepared the editorial revisions of Recommended Practice D 1729, recommended the withdrawal of Method D 406, and the editorial revision of Method D 307 to bring it up to date. The subcommittee has asked for the assistance of Committee D-20 on Plastics in an effort to combine Methods D 307 and D 791 on spectrophotometry. A method on yellowness is being prepared and work is continuing on haze and gloss measurements and on a consolidated method for color difference measurements designed to replace ultimately the present five individual methods.

Subcommittee XI on Resins (M. H. Swann, chairman), prepared the revision of Methods D 1259 and the proposed Tentative Method of Test for Total Chlorine Content of Epoxy Resins." The subcommittee recommends the adoption as standard of Method D 1651, and the continuation as tentative of Method D 1652 pending revision of this test. A study is being made of available analytical methods for identifying drying oils in alkyds; isolating polyols from alkyds for identification; and determining hydroxyl value of alkyd resins. Also, an investigation is under way of the feasibility of preparing standard alkyd resins suitable for use in calculating and expressing "solvency" of paint solvents.

Subcommittee XII on Latex Paints (P. T. Howard, chairman) prepared the proposed Tentative Method of Reporting Paint Film Failures Characteristic of Latex Paints, Specification for Asbestos-Cement Shingle Blanks to be Used as Panels in Weathering Tests of Latex and Emulsion Paints, and Test for Package Stability of Latex Paint. Work is continuing on the development of methods for determining scrub resistance, freezing-and-thawing stability, package stability, resistance to attack in the container by micro-organisms, and the development of a standard heavily chalked surface for testing the adhesion of exterior latex paints.

Subcommittee XIV on Statistical Application (M. P. Morse, chairman) has prepared a draft of a recommended practive for designing cooperative tests to investigate testing methods. The recommended procedure outlines the steps that a working group should take in evaluating a proposed method for accuracy, sensitivity, and precision. Also, the subcommittee has prepared recommended procedures for analyzing cooperative test data, has drafted definitions of statistical terms involved in precision, and has defined the types of precision and procedures for calculating precisions. These are being submitted to Committee E-11 on Quality Control of Materials for review.

Subcommittee XV on Specifications for Pigments, Dry and in Oil (C. L. Crockett, chairman) prepared the revision of Specification D 80 and recommends that the Tentative Specification D 1648 be adopted as standard. It is preparing a new specification for molybdate orange.

Subcommittee XVII on Flash Point (H. E. Riley, chairman) prepared the revision of the scope of Method D 56 that complies with similar action being taken by Committee D-2 on Petroleum Products and Lubricants. The subcommittee recommends that Method D 1310 be continued as tentative pending completion of the revision of this test. The investigation of methods for determining flash point and of defining degree of hazard used by other organizations has been completed.

Subcommittee XVIII on Physical Properties of Materials (J. P. McGuigan, chairman) has prepared a preliminary draft of a method providing for both Knoop and Pfund hardness test that will ultimately replace Method D 1474. Work is in progress on a proposed method of test for elongation and tensile strength of free films of paint, varnish, lacquer, and related products and a proposed method for rheological properties of nonnewtonian materials (thixotropic paints);

also a study is being made of a method for determining levelling characteristics of paint, presented by the New York Society for Paint Technology. Work is also continuing on the investigation of the Baker-Patton settling gage.

Subcommittee XIX on Putty, Glazing, and Caulking Compounds (H. L. Kelfer, chairman) has in preparation a method of test for initial and ultimate set of caulking compounds. Also, the subcommittee is trying to adapt the Baker-Patton settling gage for the package stability test. In connection with the test for workability, a special caulking gun with Garvey die attached has been assembled and it is hoped that some correlation will be shown between Garvey die extrusion and actual workability.

Subcommittee XXV on Lacquer (R. F. Buller, chairman) is cooperating with Subcommittee VII in the preparation of a tentative method of test for perspiration resistance of organic coatings. The subcommittee has made substantial progress in the development of methods of test for print resistance of wood lacquers, migration of plasticizer from vinyl fabrics into lacquer films, light stability tests of lacquer, and a freeze-shake test for evaluating shelf stability of multicolor lacquers.

Subcommittee XXIX on Painting of Metals (A. J. Eickhoff, chairman) is continuing its work on the procedure for the preparation of galvanized surfaces for painting, and on a method of classifying ferrous surfaces for painting. In reference to the latter, the results of a questionnaire to the steel construction industry indicated a preference for color pictures as standards rather than stereo transparencies.

This report has been submitted to letter ballot of the committee, which consists of 373 voting members; 202 members returned their ballots, of whom 173 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. T. PEARCE,
Chairman.

C. A. LOMINSKA,
Secretary.

REPORT OF COMMITTEE D-2
ON
PETROLEUM PRODUCTS AND LUBRICANTS*

This report contains the recommendations of the subordinate groups of Committee D-2 on Petroleum Products and Lubricants. The recommendations have been approved by Committee D-2 in accordance with the regulations of the Society.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1960 Annual Meeting, Committee D-2 presented to the Society through the Administrative Committee on Standards the recommendation for revisions of the following three tentatives:

Tentative Specifications for Fuel Oils (D 396 - 48 T),
Tentative Classification of Diesel Fuel Oils (D 975 - 59 T), and
Tentative Method of Test for Thermal Stability of Aviation Turbine Fuels (D 1660 - 59 T).

All three recommendations were accepted by the Standards Committee on September 29, 1960, and the revised tentatives appear in the 1960 Supplement to Book of ASTM Standards, Part 7, and in the compilation of "ASTM Standards on Petroleum Products and Lubricants," Oct., 1960, Vol. I.

PROPOSED METHODS TO BE PUBLISHED AS INFORMATION

The following proposed methods are recommended for publication as information only:[1]

Appendix I. Proposed Method of Test for Separation of Tetraethyllead and Tetramethyllead in Gasoline.
Appendix II. Proposed Method of Test for Residues in Liquefied Petroleum Gases (End Point Index Method).
Appendix III. Proposed List of Methods for Use in Specifications of Commercial Butane-Butene Mixtures.
Appendix IV. Proposed Method of Test for Knock Characteristics of Motor Fuels (The Research Method Equipped with a Split-Head Cylinder Engine).
Appendix V. Proposed Method of Sampling Turbine Engine Fuel for Cleanliness Evaluation.
Appendix VI. Proposed Method of Test for Barium, Calcium, and Zinc in Lubricating Oils and Additives (Complexometric Titration).
Appendix VII. Proposed Method of Test for Hydrocarbon Types in Low-Olefinic Gasolines by Mass Spectroscopy (a revision of Appendix VII, 1958 Report of Committee D-2).
Appendix VIII. Proposed Method of Test for Hydrocarbon Types in Olefinic Gasolines by Mass Spectrometry.
Appendix IX. Proposed Centrifuge Method for Preparing Specimen Grids for Use in the Study of Diesel Fuels by Electron Microscopy (a Revision of Appendix VII, 1960 Report of Committee D-2).
Appendix X. Proposed Method of Test for Solid Point of Aviation Turbine Fuels.
Appendix XI. Proposed Method of Test for Vapor Pressure of Petroleum Products (Micro Method).
Appendix XII. Proposed Method of Test for Shear Stability of Polymer-Thickened Oils.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] The proposed methods appear in the compilation of ASTM Standards on Petroleum Products and Lubricants, November, 1961, Vol. 1.

Appendix XIII. Proposed Method of Test for Knock Characteristics of Motor Fuels (The 1170 Severity Method).

Appendix XIV.—Proposed Method of Analysis of Natural Gas by Gas Chromatography.

Appendix XV.—Proposed Rapid Method of Test for Oxidation Stability of Steam-Turbine Oils by Rotating Bomb.

Appendix XVI.—Proposed Method for Determination of the Stability of Distillate Fuel Oils.

Appendix XVII.—Proposed Method of Test for Particulate Matter in Aviation Turbine Fuels.

Appendix XVIII.—Three Proposed Methods for Analysis of Rubber Extender Oils and Results of Cross-Test Program; "Rostler," "Texas-U.S. Chemical Co.," and "Clay-Gel."

NEW TENTATIVES

The committee recommends for publication as tentative the following specifications and methods as appended hereto:[2]

Tentative Specifications for:

Commercial Hexanes, and
Liquefied Petroleum (LP) Gases.

Tentative Methods of Test for:

Volatility of Liquefied Petroleum (LP) Gases,
Copper Strip Corrosion by Liquefied Petroleum (LP) Gases,
Roll Stability of Lubricating Grease,
Peroxide Number of Petroleum Wax,
Odor of Petroleum Wax, as published in Appendix XIII, 1957 Report of Committee D-2, 1957 compilation of "ASTM Standards on Petroleum Products and Lubricants," p. 1051.
20-deg Specular Gloss of Waxed Paper, as published in Appendix III, 1957 Report of Committee D-2, 1957 compilation of "ASTM Standards on Petroleum Products and Lubricants," p. 979 with revision as follows:

Section 4.—Add the following new Section 4 entitled "Significance" renumbering present Section 4 and subsequent sections accordingly:

4. This method gives good correlation with visual gloss or shininess provided the sample is flat. The degree of correlation decreases as the sample deviates from being flat.

Amyl Nitrate in Diesel Fuels,
Naphthalene Hydrocarbons in Aviation Turbine Fuels by Ultraviolet Spectrophotometry, as published in Appendix III, 1959 Report of Committee D-2, 1959 compilation of "ASTM Standards on Petroleum Products and Lubricants," p. 1135, with the following revision:

Section 11.—Add the following new Section 11 on Precision.

11. The following criteria should be used for judging the acceptability of results (95 per cent probability):

(*a*) *Repeatability.*—Duplicate results by the same operator should not be considered suspect unless they differ by more than 0.05.

(*b*) *Reproducibility.*—The results submitted by each of two laboratories should not be considered suspect unless the two results differ by more than 0.11.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

D 381 – 58 T,[3] Tentative Method of Test for Existent Gum in Fuels by Jet Evaporation:

Section 4.—Add a new Section 4 on Significance as follows, renumbering the present Section 4 and subsequent sections and references thereto accordingly:

4. The true significance of this method for determining gum in motor gasoline is not firmly established. Certainly it has been proved that high gum can cause induction-system deposits and sticking of intake valves and in most instances it can be assumed that low gum will insure absence of induction-system difficulties. The user should, however, realize that the test is not of itself correlative to induction system deposits. The primary purpose of the test, as applied to motor gasoline, is the measurement of the oxidation products formed in the sample prior to or during the comparatively mild conditions of the test procedure. Since many motor gasolines are purposely blended with nonvolatile oils or additives, the heptane extraction step is necessary to remove these from the evaporation residue so that the deleterious material, gum, may be determined.

Note 2.—Renumber as Note 1.

Section 6(b).—Change "311 F (155 C)" in line 3 to "324 F (162 C)."

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[2] The new tentatives appear in the 1961 Book of ASTM Standards, Part 7.

[3] 1958 Book of ASTM Standards, Part 7.

Section 8(f).—At the end of the second sentence add "(Note 2)."

New Note.—After Section 8(*f*) add a new Note 2 to read as follows:

NOTE 2.—Qualitative evidence of motor gasoline contamination may be obtained by weighing the residue at this point if retained samples of the original finished gasoline are available for reference testing. This reference testing is essential since motor gasoline may contain deliberately added materials that are nonvolatile. If evidence of contamination is obtained, further investigation is indicated.

Section 4.—Renumber as Section 5, and reletter the paragraphs as follows:

Present Paragraph	Relettered Paragraph
(*a*)	(*d*)
(*b*)	(*e*)
(*c*)	(*g*)
(*d*)	(*b*)
(*e*)	(*c*)
(*f*)	(*a*)
(*g*)	(*h*)
(*h*)	(*f*)

Section 5.—Renumber as Section 6, and reletter Paragraphs (*b*), (*c*), and (*d*) as (*d*), (*b*), and (*c*), respectively.

D 396 - 60 T,[4] Tentative Specifications for Fuel Oils:

Table I.—For grade No. 2 fuel oil, change Gravity, deg API from "26" to "30[g]."

New Footnote g.—Add a new Footnote (*g*) to read as follows:

[g] In the states of Alaska, Arizona, California, Hawaii, Idaho, Nevada, Oregon, Utah, and Washington, a minimum gravity of 28 deg API is permissible.

D 483 - 60 T,[4] Tentative Method of Test for Unsulfonated Residue of Petroleum Plant Spray Oils:

New Section 6, Note 2, and Section 7.—After Section 5, insert the following new Section 6, new Note 2, and new Section 7, renumbering subsequent sections and notes accordingly:

6. *Reference Spray Oil.*—Test results for unsulfonated residue are highly dependent upon rate of shaking. A reference spray oil has been calibrated for unsulfonated residue by a group of cooperating laboratories using both machine and hand shaking. Instructions are given in Section 7 for using this reference oil as a guide to ensure that the rate of shaking is correct.

NOTE 2.—The reference spray oil may be obtained by addressing a request to Chief, Bureau of Chemistry, California Department of Agriculture, 1220 N St., Sacramento 14, Calif. or from the chairman of Subcommittee XVII of ASTM Committee D-2.

7. *Calibration.*—(*a*) *Machine Shaking Rate.*—A rate of 425 cycles per min was used in the cooperative work to establish the unsulfonated residue of the reference spray oil. There are small variations in severity of shaking between individual machines, even when they are newly built and of the same make; and these differences may increase with use. Consequently, each laboratory should run occasional tests on the reference spray oil. If an unsulfonated residue is found which differs by more than ±0.4 per cent from the established value, the rate of shaking should be adjusted accordingly. A faster rate tends to give a lower unsulfonated residue, and vice versa.

(*b*) *Hand Shaking Rate.*—Each operator should standardize his technique of shaking so as to obtain the established value (within ±0.4 per cent) on the reference spray oil. A rate of 300 cycles per min was used in the manual shaking tests in the cooperative program on the reference oil; however, an individual operator's calibrated rate may differ appreciably from 300 cycles.

Section 6.—Renumber as Section 8, and revise Paragraph (*a*) to read:

8. (*a*) Adjust the temperature of the boiling-water bath to 99.5 to 100 C and keep it in this temperature range throughout the test. Adjust the shaker rate and check it before and after each test to make sure that the rate does not deviate more than ±10 cycles per min from the rate established by calibration.

Renumber Note 2 as Note 3 and revise the last sentence to read: "Shake at the rate established by calibration within a tolerance of ± 10 cycles per min."

In Paragraph (*e*) change the second sentence to read: "Shake for 10 sec at the rate established in the calibration with the reference spray oil (**Caution,** see Note 4)."

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[4] 1960 Supplement to Book of ASTM Standards, Part 7.

Renumber Note 3 as Note 4 and change the last sentence to read: "Substitute the counter for the timer and shake a total number of cycles equal to one-sixth of the number representing the established rate in cycles per minute, even though it requires more than 10 sec (for example, if the rate is 425 cycles per min, shake 71 cycles)."

In Paragraph (*g*), change the latter portion of the last sentence to read: "...and point *B* refers to the lowest portion of the interface between the clear oil and sulfonation acid."

Figure 1.—Delete the dotted line above and below the interface at *A* and above the interface at *B*.

Section 8.—Renumber as Section 10, and revise the tables to read:

Range of Unsulfonated Residue, per cent	Repeatability (Machine Shaking)
85 to 96	0.5

Range of Unsulfonated Residue, per cent	Reproducibility (Machine Shaking)
85 to 96	2.0

Footnote 3.—Delete this footnote, and reference thereto.

Appendix.—In the second sentence of Paragraph A3, delete the portion of the sentence after "... 1⅛ in." and insert at this point a sentence reading: "The machine shall be capable of quickly reaching, and maintaining for 10 sec, any required rate of shaking in the range of 300 to 500 cycles per min, within a tolerance of ±10 cycles."

Temperatures.—Change all Fahrenheit temperatures throughout the method to Centigrade (Celsius) temperatures as follows:

Present Temperature Designations	Proposed
212 F	100 C
77 F	25 C
211 to 212 F	99.5 to 100 C
77 ± 1 F	25 ± 0.5 C

D 613 – 59 T,[5] Tentative Method of Test for Ignition Quality of Diesel Fuels by the Cetane Method:

Section 9.—Change to read:

9. Two meters, the Waukesha Transistorized Time Lag Meter[4] and the Ignition Delay Meter, Model ECS-1[4a] are approved for measuring ignition delay by this method. The Waukesha Transistorized Time Lag Meter has preferred status. Both meters have three position switches marked CALIBRATE, INJECTION ADVANCE, and IGNITION DELAY, and controls for making basic calibration settings. The three position switch and calibration controls on both meters are quite similar and the same set of operating instructions are applicable for either meter. Detailed instructions for calibration and use of either meter are given in Appendix III on Operation, Appendix VII on Instrumentation, and in the following paragraphs.

In Paragraph (*a*), change the last part of the first sentence to read: "... and turn on the power switch to the meter."

Footnotes.—Renumber Footnote 4 as 4*a*, and add a new Footnote 4 to read: "[4] Manufactured and available from the Waukesha Motor Co., Waukesha, Wis."

Section 703(c).—Change to read:

(*c*) *Injector Pickup* (*Green Band*):

(*1*) Two mountings, one external and the other internal, are approved for the injection pickup used for the ECS-1 meter. For the external mounting, which is the preferred type, set the gap between the end of the pickup and spindle to 0.060 in. and lock in this position. For the internal type, set the pickup gap by screwing the pickup into the body until the pole piece just touches the end of the spindle. Then, unscrew the pickup 1½ turns from this point of contact and lock in this position. The recommended pickup gap settings for the externally and internally mounted pickups are nominal and slight adjustments may be required to obtain satisfactory operation with different combinations of meters and pickups. When maintenance or adjustment of injection pressure is required with either type of pickup mounting, loosen the locknut and unscrew the pickup to prevent the possibility of it being damaged by tightening it against the end of the spindle. Also, the use of an insufficient pickup gap for operation will result in a bent or battered pickup core and unsatisfactory operation.

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[5] See ASTM Manual for Rating Diesel Fuels by the Cetane Method, 1959.

(*2*) Only one mounting, which is of the external type is approved for the injection pickup of the Transistorized Delay Meter. Set the gap between the end of this pickup and spindle to 0.040 in. and lock in this position. The recommended gap setting of 0.040 in. for this pickup is nominal and slight adjustments may be required to obtain satisfactory operation with different combinations of meters and pickups. When maintenance or adjustment of injection pressure is required, loosen the locknut and unscrew the pickup to prevent the possibility of it being damaged by tightening it against the end of the spindle. Also, the use of an insufficient pickup gap will result in a bent or battered pickup core and unsatisfactory operation.

D 1319 – 60 T,[4] Tentative Method of Test for Hydrocarbon Types in Liquid Petroleum Products by Fluorescent Indicator Adsorption:

Section 1.—Delete the word "debutanized" in the first sentence. Delete the third sentence. Change the last sentence to read: "Samples should be depentanized if they contain any of the following: C_3 or lighter hydrocarbons; more than 5 per cent C_4 hydrocarbons; more than 10 per cent C_4 and C_5 hydrocarbons."

Figure 1.—In the figure, change "Compressed Air" to "Pressuring Gas"; "Gel added after sample" to "Pack gel to this level." Delete "Pack Gel to this Level Initially" and arrows; and delete dimensions "15" and "40" in charger sections of both columns and replace with "55."

Section 3.—In the second sentence change "air" to "gas."

In the second sentence of Paragraph (*d*) delete phrase "as used by barbers."

Section 4(*b*).—Delete this paragraph and reletter subsequent paragraphs.

Footnote 7.—Delete this footnote and renumber subsequent footnotes.

New Section.—Add a new Section 5 to read as follows, renumbering present Section 5 and subsequent sections accordingly:

5. ***Preparation of Sample.***—(*a*) *Samples Containing Light Hydrocarbons.*—Samples should be depentanized (Note 4) if they contain any of the following: C_3 or lighter hydrocarbons; more than 5 per cent C_4 hydrocarbons; more than 10 per cent C_4 and C_5 hydrocarbons.

NOTE 4.—Proposed Method of Test for Depentanization of Gasoline and Naphthas was published for information as Appendix IV, of the compilation "ASTM Standards on Petroleum Products and Lubricants," October, 1960, Vol. I.

(*b*) *Samples Containing Colored or Fluorescing Substances.*—If difficulties in reading zone boundaries are encountered with samples containing substances that are dark colored or that fluoresce under ultraviolet light, removing the interfering substances (for example, by distillation) may be helpful.

Present Section 5.—Renumber as Section 6 and in the third sentence of Paragraph (*a*), delete the words "specified 100- to 200-mesh." Change the last part of the fourth sentence to read: "or tap along the length of the column to pack the gel firmly." Delete the parenthetical seventh sentence. Change the last sentence to read: "Add more of the silica gel until the tightly packed gel extends approximately 55 mm into the charger section (a 5-min vibration period is usually satisfactory when the vibrator is used)."

In Paragraph (*b*), change "air" to "gas" and delete the phrase "as required for the true-bore column."

In Paragraph (*c*), connect sentences 2 and 3 by a semicolon, and make it the first sentence. Make the present first sentence the second sentence.

In Paragraph (*d*), replace the first four sentences with: "Chill the sample and a 1-ml hypodermic syringe to 35 to 40 F. Draw 0.75 ± 0.03 ml of sample into the syringe and inject the sample 30 mm below the surface of the gel in the charger section."

Delete Notes 5 and 6. Renumber Note 4 as 5 and Notes 7 and 8 as 6 and 7.

In the first sentence of Paragraph (*h*) change "air" to "gas."

D 1321 – 60 T,[4] Tentative Method of Test for Needle Penetration of Petroleum Waxes:

Section 1.—In the last sentence, change the phrase "... no greater than 150" to "... of not greater than 250."

D 1405 – 57 T,[3] Tentative Method of Test for Estimation of Net Heat of Combustion of Liquid Petroleum Products:

Section 1.—To the table in Note 2, add the turbine fuel specification: "ASTM D 1655. ... Type A and A-1 Turbine Fuels."

Section 2(a).—Add kerosine and Type A and A-1 aviation turbine fuels to the table of equations, as follows:

Type Fuel	Equation	Equation No.
Kerosine, Type A or A-1 (D 1655)..	Q_p (net) = 17 919 + 0.10923 ($A \times G$)	(4)

Renumber Eq 4 as 5.

Tables I and IA.—Change equation numbers in the title from "1 and 4" to "1 and 5."

Footnote 4.—Change last three lines to read: "... Other Properties," *Chemical and Engineering Data Series* Vol. 3, pp. 20–28 (1958)."

New Footnote 5.—Add a new footnote 5 referenced to the first word in section 2 (*a*), to read: "[5] L. Fano, S. Marantz, T. W. Mears, J. A. Walker, G. T. Armstrong, and R. S. Jessup, National Bureau of Standards, Washington, D. C., private communication."

Section 2(c).—In the first sentence, add "3 and 4" to the equations. To the last sentence add the phrase "and to ASTM Specifications D 1655, Types A and A-1."

Section 4(b).—To the last sentence, add the phrase "and Table IV should be used for kerosine-type jet fuels (ASTM Specifications D 1655, Types A and A-1)."

Table II.—Change equation numbers in the title from "2 and 4" to "2 and 5."

Table III.—Change equation numbers in the title from "3 and 4" to "3 and 5."

Table IV.—Add a new Table IV, as shown in the accompanying Table I.[6]

D 1655 – 59 T,[7] Tentative Specifications for Aviation Turbine Fuels:

Table I.—Replace with the table shown in the accompanying Table II.[8]

Section 9.—Add Paragraphs (*u*) and (*v*) to read:

> (*u*) *Luminometer Number.*—Method of Test for Luminometer Numbers of Aviation Turbine Fuels (ASTM Designation; D 1740).
>
> (*v*) *Naphthalene Content.*—Method of Test for Naphthalene Hydrocarbons in Aviation Turbine Fuel by Ultraviolet Spectrophotometry (ASTM Designation: D 1840).

D 1656 – 60 T,[9] Tentative Method of Test for Knock Characteristics of Motor Fuels Above 100 Octane Number by the Research Method:

Section 6(k).—Add the following to the paragraph:

> It also is permissible to vary intake air temperature as described in Section 10(*b*) to obtain the calibrated values on the toluene standardization fuel blends. If this is done, the intake air temperature shall be maintained within ±2 F (±1.1 C) of the temperature at which the calibrated rating is obtained on the toluene standardization fuel blend used.

Section 6(o).—Change to read as follows:

> (*o*) *Standard Knock Intensity,* is defined as the level of knock obtained on a blend of tetra-

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[6] Table I is not included in this report but appears in the revised tentative; see 1961 Book of ASTM Standards, Part 7.

[7] 1959 Supplement to Book of ASTM Standards, Part 7.

[8] Table II is not included in this report but appears in the revised tentative; see 1961 Book of ASTM Standards, Part 7.

[9] See ASTM Manual for Rating Motor Fuels by Motor and Research Methods 1960.

ethyllead in *iso*octane at the specified operating conditions of this method and at the micrometer reading shown in Table VIII or IX, corrected for the prevailing barometric pressure in accordance with Table X, for the antiknock level of the leaded *iso*octane blend used.

Section 8.—Change second sentence to read as follows:

Unless the intake air temperature is varied in order to obtain the calibrated ratings on the toluene standardization fuel blends as described in Section 10(*b*), it shall be maintained at the value specified for the prevailing barometric pressure in Table X.

Section 10.—Add a new Paragraph (*b*) to read as follows:

(*b*) It is permissible to vary the intake air temperature to obtain the calibrated rating on the 103.3 octane-number toluene standardization fuel or the other toluene standardization fuel blends shown in Section 202 provided, (*1*) the initial rating on the toluene standardization fuel is obtained at the intake-air temperature shown in Table X, (*2*) the cylinder height used for this rating is in exact agreement with the micrometer setting shown in Tables VIII or IX, corrected for the prevailing barometric pressure in accordance with Table X, for the calibrated rating of the toluene standardization fuel blend used, (*3*) the rating obtained at the conditions specified in (*1*) and (*2*) is within the octane-number limits shown in the following table, and (*4*) the cylinder height used in (*2*) is maintained for tests on the toluene standardization fuel blend at other intake-air temperatures:

Toluene Standardization Fuel Blend				Octane Number Limits	
Toluene, per cent	Heptane, per cent	*Iso*-octane, per cent	Calibrated Research Octane Number	Min	Max
74	11	15	103.3	102.1	104.5
74	6	20	108.0	106.3	109.7
74	...	26	113.7	111.5	115.9

The limits of testing range for the intake air temperature determined by this method are approximately ±3 octane numbers from the calibrated value of the toluene standardization fuel blend used. As an example of this requirement, the intake air temperature determined to give the calibrated rating of 103.3 on this toluene standardization fuel blend within the limits of ±2 F (1.1 C) would have application over an approximate testing range from 100.3 to 106.3 octane number.

Section 10(*b*).—Reletter as (*c*) and change to read as follows:

(*c*) For subsequent testing of samples at the intake air temperature specified in Table X or at the adjusted intake air temperature described in Paragraph (*b*), adjust the cylinder heights for standard knock intensity, using the knockmeter as a guide, provided no change has been made in the detonation meter adjustment in the meantime. Make certain that the cylinder heights as measured by micrometer settings are within ±0.010 in. of those given in guide Table VIII or IX, corrected from Table X for the prevailing barometric pressure, for the antiknock level of the reference fuel blend that matches the sample. When the engine is being operated at the intake air temperatures shown in Table X, readjust the intake air temperature for agreement with the values shown in this table if there are significant changes in barometric pressure during an operational period. Also, if there are significant changes in barometric pressure when an engine has been standardized in accordance with Paragraph (*b*), it is necessary that the operations in that paragraph be repeated.

Section 10(*c*).—Reletter as (*d*).

Section 112(*a*).—Change fifth sentence to read as follows:

The carburetor is fitted with a 90-deg elbow and vertical tube connecting with the surge tank. The 90-deg elbow is preferred equipment; however a curved intake pipe may be used if desired.

D 1660 – 60 T,[7] Tentative Method of Test for Thermal Stability of Aviation Turbine Fuels:

Appendix I, Section A6(*g*).—Change to read as follows:

(*g*) The thermometry system including thermocouples, leads, and temperature indicators shall be calibrated upon control set-up of the apparatus and whenever there is doubt as to its accuracy. Calibration is accomplished with a dual preheater thermocouple by connecting one lead to a standard laboratory potentiometer and connecting the other lead to the temperature indicator. The temperature calibration is made with fuel flowing at the desired test temperature. The temperature indicator should then be adjusted to read at true temperature.

Appendix I, Section A11.—Change the second sentence to read as follows:

Calibrate the automatic temperature controllers to indicate true temperature by connecting the dual preheater thermocouple to a potentiometer, and by adjusting the green indicating pointer to the true temperature.

Appendix II.—Add a new Section B26 to read as follows:

B26.—*Preheater.*—*Dual Thermocouple.*—The use of dual thermocouples[13] is specified for calibration of the temperature indicated by means of a potentiometer.

D 1717 - 60 T,[4] Tentative Method for Analysis of Commercial Butane-Butene Mixtures (Gas Chromatography Method):

Title.—Change to read: "Analysis of Certain Light Hydrocarbons by Gas Chromatography."

Section 1.—Change to read as follows:

1. This method is applicable to the analysis by gas chromatography of:

(*1*) Commercial butane-butene (B-B mix),

(*2*) Commercial isobutylene of at least 98 per cent.[3]

Section 2.—Delete the second sentence and add: "For B-B mixtures the composition of the sample is determined from the chromatogram by comparing peak heights or areas with those obtained from a standard sample. The composition of high-purity samples (98 per cent +) is determined from the chromatograms by measuring the area under the peaks."

Footnote 3.—Add a new Footnote 3 to read as follows, renumbering present footnotes 3 and 4 as 4 and 5:

[3] This procedure may be applicable to other C_2 to C_4 products, but commercial butane-butene mixtures and high-purity isobutylene are the only products that have been cooperatively studied by the sponsoring committees.

Section. 4—Revise Paragraph (*e*) to read as follows:

(*e*) *Synthetic Blend.*—Prepare a synthetic mixture from pure hydrocarbons that is near the composition of the sample to be analyzed. The mixture should be accurate to 0.1 per cent of any component. A synthetic mixture is not necessary when analyzing high-purity samples.

Footnote 6.—Delete this footnote and add a new Footnote 6 to read as follows:

[6] A suitable calibration standard for B-B mix may be obtained from the Special Products Div., Phillips Petroleum Co., Bartlesville, Okla.

Section 6.—Letter as "(*a*) *B-B Mixtures*" and retain the present Note 2. Add a new Paragaph (*b*) to read as follows:

(*b*) *High-Purity Samples.*—It is not necessary to use a synthetic mixture when analyzing high-purity samples. Liquefied samples should be withdrawn from the liquid phase and tested in accordance with Paragraph (*a*). Totally gaseous samples will necessarily have to be charged from the gas phase.

Section 7.—Add a new Paragraph (*c*) to read as follows:

(*c*) *High-Purity Mixtures, Area Method.*—Measure the area of each component, and assume the area to be directly proportional to the concentration of that component.

Section 8.—Change to read as follows:

8. Normalize the values obtained and report the values obtained on B-B mix to the nearest 0.1 per cent and to the nearest 0.01 per cent on high-purity products.

Section 9.—In Paragraph (*a*), replace the present repeatability results with the following:

Component, per cent by weight	B-B Mix, Repeatability	High-Purity, Repeatability
0 to 5..........	0.1	
5 to 25.........	0.3	Minor compounds, 0.04
Above 25.......	0.5	Major compounds, 0.07

In Paragraph (*b*), replace the present reproducibility results with the following:

Component, per cent by weight	B-B Mix, Reproducibility	High-Purity, Reproducibility
0 to 5..........	0.3	Minor compounds, 0.07
5 to 25.........	0.9	
Above 25.......	1.5	Major compounds, 0.20

WITHDRAWAL OF STANDARD

The committee recommends the withdrawal of the following standard because of lack of use:

Standard Method of Analysis of 60 Octane Number *Iso*octane - Normal Heptane ASTM Knock Test Reference Fuel Blends by Infrared Spectrophotometry (**D 1095 - 54**).

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentative methods be approved for reference to letter ballot of the Society for adoption as standards without revision:

D 93 - 58 T, Flash Point by Pensky-Martens Closed Tester,[3]

D 1159 - 59 T, Bromine Number of Petroleum Distillates by Electrometric Titration,[7]

D 1160 - 57 T, Distillation at Reduced Pressure of Petroleum Products,[3]

D 1218 - 52 T, Measurement of Refractive Index and Refractive Dispersion of Hydrocarbon Liquids,[3]

D 1263 - 53 T, Leakage Tendencies of Automotive Wheel Bearing Greases,[3]

ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that the Tentative Method of Sampling Petroleum and Petroleum Products, (**D 270 - 55 T**) be approved for reference to letter ballot of the Society for adoption as standard revised, as appended hereto.[10]

REVERSION OF STANDARD TO TENTATIVE WITHOUT CHANGE

The committee recommends that the Standard Method of Test for Oxygen in Light Hydrocarbon Vapors (**D 1021 - 55**)[3] be reverted to tentative without change.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

D 56 - 56,[11] Standard Method of Test for Flash Point by Tag Closed Tester (Jointly with Committee D-1):

Section 1.—Change to read as follows:

1. This method of test is intended for determining the flash point of all mobile liquids flashing below 175 F (79 C), except products classed as fuel oil.

Appendix II, Section A6.—Change the purity statement for xylene to read as follows:

Freezing point....11.23 C, min (95 per cent molal purity) as determined by ASTM Method D 1015, Test for Freezing Points of High-Purity Hydrocarbons.

D 86 - 59,[12] Standard Method of Test for Distillation of Petroleum Products:

Section 6(d).—Change the second sentence to read as follows:

Measure 100 ml of the sample in the graduated cylinder and transfer it as completely as practicable to the distillation flask.

Section 6(f).—Delete "100-ml" from the first sentence.

Section 8(h).—In the first sentence, change "linear interpolation procedure" to read: "arithmetical procedure."

Replace Paragraph (*1*) with the following:

(*1*) *Graphical Procedure.*—Using graph paper with uniform subdivisions, plot each thermometer reading (corrected for barometric pressure, if required) against its corresponding per cent recovered. Plot the initial boiling point at zero per cent recovered. Draw a smooth curve connecting the points. For each prescribed per cent evaporated, deduct the distillation loss, in order to obtain the corresponding per cent recovered, and take from the graph the thermometer reading which this per cent recovered indicates (Note 9). Values obtained by graphical interpolation procedures are affected by the care with which the plot is made.

Change the heading of Paragraph (*2*) to read: "Arithmetical Procedure."

Note 9.—In the first sentence change

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[10] The revised method appears in the 1961 Book of ASTM Standards, Part 7.

[11] 1958 Book of ASTM Standards, Parts 7 and 8.

[12] 1959 Supplement to Book of ASTM Standards, Part 7; 1958 Book, Part 8.

"linear interpolation" to read: "arithmetical procedure."

New Note 10.—Immediately after the present Note 9, add a new Note 10, to read as follows:

NOTE 10.—See the Appendix for numerical examples illustrating the foregoing graphical and arithmetical procedures.

Appendix.—Add an Appendix to read as follows:[13]

New Figure 2.—Add a Fig. 2 as shown in the accompanying Fig. 1.[14]

D 91 – 52,[3] Standard Method of Test for Precipitation Number of Lubricating Oils:

Figure 1.—Revise as shown in the accompanying Fig. 2.[15]

Table I.—Replace with the table shown in the accompanying Table III.[15]

Section 7.—Change to read as follows:

7. The following criteria should be used for judging the acceptability of results (95 per cent probability):

(*a*) *Repeatability.*—Duplicate results by the same operator should not be considered suspect unless they differ by more than the following amount:

Precipitation Number	
0.00 to 1.20	10 per cent of mean

(*b*) *Reproducibility.*—The result submitted by each of two laboratories should not be considered suspect unless the two results differ by more than the following amount:

Precipitation Number	
0.00 to 1.20	30 per cent of mean

D 95 – 58,[16] Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials:

This revision has been suggested by ASTM Committee D-16 on Industrial Aromatic Hydrocarbons and is subject to approval by both Committee D-2 and Committee D-4.

Section 1.—To the last sentence add the following words: "and crude or refined tar acids."

New Footnote.—Add a new Footnote 4, referenced to the word "trap" in Section 3(*a*) to read:

"[4] For crude or refined tar acids, a 2000-ml vessel is used."

D 128 – 59,[7] Standard Methods of Analysis of Lubricating Grease:

Sections 22 to 27.—Delete and renumber subsequent sections and notes accordingly, substituting the following for the material deleted under the heading "Water."

22. (*a*) Determine water in accordance with the Method of Test for Water in Petroleum Products and Other Bituminous Materials (ASTM Designation: D 95).

(*b*) For solvent, use the petroleum distillate as described in Section 4(*b*) of Method D 95.

(*c*) In accordance with Section 7 of Method D 95, calculate and report water as per cent by weight.

D 189 – 58,[3] Standard Method of Test for Conradson Carbon Residue of Petroleum Products:

Revise as appended hereto.[17]

D 288 – 57,[3] Standard Definitions of Terms Relating to Petroleum:

Add the following definitions:

Petroleum Wax.—A product separated from petroleum which is solid or semisolid at 77 F and consists essentially of a mixture of saturated hydrocarbons.

Petrolatum.—A petroleum wax of unctuous structure containing substantial amounts of oil.

D 357 – 60,[7] Standard Method of Test for Knock Characteristics of Motor Fuels Below 100 Octane Number by the Motor Method:

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[13] This Appendix is not included in this report but appears in the revised standard; see 1961 Book of ASTM Standards, Part 7.

[14] Figure 1 is not included in this report but appears in the revised standard; see 1961 Book of ASTM Standards, Part 7.

[15] Figure 2 and Table III are not included in this report but appear in the revised standard; see 1961 Book of ASTM Standards, Part 7.

[16] 1958 Book of ASTM Standards, Parts 4, 7 and 8.

[17] The revised standards appear in the 1961 Book of ASTM Standards, Part 7.

Section 112(a).—Change fifth sentence to read: "The carburetor is fitted with a 90-deg elbow and vertical tube connecting with the surge tank. The 90-deg elbow is preferred equipment; however a curved intake pipe may be used if desired."

D 445 - 60,[4] Standard Method of Test for Kinematic Viscosity:

New Section 8.—Add the following new Section 8 renumbering the present Sections 8 and 9 as 9 and 10:

8. (*a*) Steam-refined cylinder oils, black lubricating oils, residual fuel oils, and other materials that may possibly contain waxes or resins require special handling to obtain reproducible results. In order to control the possible anomalies in testing such material, the thermal history of the sample should be controlled. This special procedure will aid in the control of wax crystals and lattice structure formations.

(*b*) Heat a homogenous representative sample in a loosely stoppered container for 1 hr in an oven maintained at 212 to 215 F.

(*c*) Immediately filter the preheated sample through a 200 mesh screen directly into the viscometer. Where direct filtration into the viscometer is not practicable, charge the viscometer in the manner dictated by the design of the instrument.

(*d*) Complete the test as described in Section 7(*f*) to (*k*).

(*e*) Complete viscosity determinations on these materials within 1 hr after filling the viscometer with the sample. Do not run repeat determinations on the same portion of these materials.

D 526 - 56,[3] Standard Method of Test for Tetraethyllead in Gasoline (Gravimetric Method):

Revise as appended hereto.[17]

D 908 - 60,[7] Standard Method of Test for Knock Characteristics of Motor Fuels Below 100 Octane Number by the Research Method:

Section 6(k).—Add the following to this paragraph:

It also is permissible to vary intake air temperature as described in Section 10(*b*) to obtain the calibrated values on the toluene standardization fuel blends. If this is done, the intake air temperature shall be maintained within ±2 F (1.1 C) of the temperature at which the calibrated rating is obtained on the toluene standardization fuel blend used.

Section 6(o).—Change to read as follows:

(*o*) ***Standard Knock Intensity,*** is defined as the level of knock obtained on a blend of *iso*octane and *n*-heptane at the specified operating conditions of this method and at the micrometer reading shown in Table V, corrected for the prevailing barometric pressure in accordance with Table VI, for the octane number of the blend used.

Section 8.—Change the second sentence to read as follows:

Unless the intake air temperature is varied in order to obtain the calibrated ratings on the toluene standardization-fuel blends as described in Section 10(*b*), it shall be maintained at the value specified for the prevailing barometric pressure in Table VI.

Section 10.—Add a new Paragraph (*b*) to read as follows:

(*b*) It is permissible to vary the intake air temperature to obtain the calibrated rating on the 93.4 octane number toluene standardization fuel or other toluene standardization-fuel blends shown in Section 202 provided (*1*) the initial rating on the toluene standardization-fuel is obtained at the intake air temperature shown in Table VI, (*2*) the cylinder height used for this rating is in exact agreement with the micrometer setting shown in Table V, corrected for the prevailing barometric pressure in accordance with Table VI, for the calibrated rating of the toluene standardization-fuel blend used, (*3*) the rating obtained at the conditions specified in (*1*) and (*2*) is within the octane number limits shown in the following table and, (*4*), the cylinder height used in (*2*) is maintained for tests on the toluene standardization-fuel blend at other intake air temperatures:

Toluene Standardization Fuel Blend				Octane Number Limits	
Toluene, per cent	Heptane, per cent	*Iso*octane, per cent,	Calibrated Research Octane Number	Min	Max
50	50	...	65.2	64.3	66.1
58	42	...	75.5	74.7	76.3
66	34	...	85.0	84.3	85.7
74	26	...	93.4	92.9	93.9
74	21	5	96.7	96.2	97.2
74	16	10	99.6	98.8	100.4

The limits of testing range for the intake air temperature determined by this method are approximately ±5 octane numbers from the calibrated value of the toluene standardization-fuel blend used. As an example of this requirement, the intake air temperature determined to give the calibrated rating of 93.4 octane number on this toluene standardization-fuel blend within the limits of ±2 F (1.1 C) would have application over an approximate testing range of 88.4 to 98.4 octane number.

Reletter present Paragraph (*b*) as (*c*), and revise to read as follows:

For subsequent testing of samples at the intake air temperature specified in Table VI or at the adjusted intake air temperature described in Paragraph (*b*), adjust the cylinder height for standard knock intensity, using the knock meter as a guide, provided no change has been made in the detonation meter adjustment in the meantime. Make certain that the cylinder heights as measured by micrometer settings are within ±0.010 in. of those given in Table V, corrected from Table VI for the prevailing barometric pressure, for the antiknock level of the reference fuel blend that matches the sample. When the engine is being operated at the intake air temperature shown in Table VI, readjust the intake air temperature for agreement with the values shown in this table if there are significant changes in barometric pressure during an operating period. Also, if there are significant changes in barometric pressure when an engine has been standardized in accordance with Paragraph (*b*), it is necessary that operations in that paragraph be repeated.

Section 112(a).—Change as shown above for Method D 357.

D 1020 - 59,[7] Standard Method of Test for Alpha Acetylenes in Light Hydrocarbons:

Title.—Change to read: "Alpha Acetylenes in Butadiene, Butadiene Concentrates, and Butane-Butene Mixtures (Silver Nitrate Method)."

Section 1.—Change the first sentence to read: "This method describes a procedure for the determination of the monoalkyl acetylenes present in polymerization grade butadiene, butadiene concentrates, and butane-butene mixtures."

Section 2.—Change to read as follows:

2. The liquid sample is volatilized and passed through a series of five absorbers. The first absorber, containing dilute sulfuric acid, removes any basic compounds present. The second absorber, containing sodium bisulfite solution, removes carbonyl compounds present. The third absorber, containing a solution of sodium hydroxide, removes sulfur dioxide, hydrogen sulfide, mercaptans, and any acidic compounds present. The fourth and fifth absorbers, containing alcoholic silver nitrate solution, reacts with the acetylene liberating acid. The total acetylene content (with the exception of dialkyl acetylenes) is determined by titrating the increased acidity of the silver nitrate solution with standard base and reported as per cent vinyl acetylene.

Section 4.—Add new Paragraphs (*f*) and (*g*) to read:

(*f*) *Sodium Hydroxide Solution (10 per cent).*—Dissolve 110 g of NaOH in 1 liter of distilled water.

(*g*) *Sodium Bisulfite Solution (25 per cent).*—Dissolve 33 g of $NaHSO_3$ in 100 ml of distilled water.

New Note.—Add a new Note 2 after Section 4(*g*) to read as follows, renumbering present Notes 2 and 3 as 3 and 4:

Note 2.—The sodium bisulfite solution should be prepared fresh daily. The frequency of changing the solution in the absorber will depend upon the number of samples run that are high in carbonyl concentration.

Section 5(a).—Change to read as follows:

5. (*a*) See Fig. 1. To the sulfuric acid gas washing bottle add about 100 ml of H_2SO_4 (1:9) and 5 drops of bromophenol blue indicator solution; replace the acid with fresh acid when the indicator shows it to be neutralized. To the sodium bisulfite bottle add about 100 ml of sodium bisulfite solution and to the sodium hydroxide bottle about 100 ml of sodium hydroxide solution (10 per cent). To each of the silver nitrate bottles add about 100 ml of the alcoholic silver nitrate solution and 8 drops of mixed indicator solution. Neutralize the solutions in both absorbers to the grayish green end point with the 0.05 *N* sodium hydroxide solution. Assemble the apparatus as shown in Fig. 1, using short lengths of neoprene tubing.

Section 5(c).—Change third sentence to read: "After the sample has completely evaporated, attach a nitrogen line to the flask and pass a slow stream of nitrogen through the system in order to displace all hydrocarbon vapors; 5 to 10 min is sufficient time to accomplish this."

Figure 1.—Change from 3 bottles in series to 5. Label the bottles as follows, starting with the one nearest the 500-ml Erlenmeyer flask and going to the right in the following order: Sulfuric Acid, Sodium Bisulfite, Sodium Hydroxide, and the last two, Alcoholic Silver Nitrate.

Section 7.—Insert above Paragraph (*a*) and below the introductory sentence the following label: "For Butadiene and Butadiene Concentrates."

Add the following to the end of the present Paragraph (*b*), the label "For Butane-Butene Mixtures and Similar Light Hydrocarbons," and add the following two paragraphs:

(*c*) *Repeatability.*—Duplicate results by the same operator should not be considered suspect unless they differ by more than the following amount:

Acetylenes, per cent by weight	Repeatability
0.00 to 0.10	0.004

(*d*) *Reproducibility.*—The result submitted by each of two laboratories should not be considered suspect unless the two results differ by more than the following amounts:

Acetylenes, per cent by weight	Reproducibility
0.00 to 0.02	0.014
0.02 to 0.10	0.025

D 1219 – 56,[3] Standard Method of Test for Mercaptan Sulfur in Aviation Turbine Fuels (Color-Indicator Method:)

Section 1.—Change Section 1 and Note 1 to read as follows:

1. This method describes a procedure for the determination of mercaptan sulfur in aviation turbine fuels containing from 0.0003 to 0.01 per cent by weight of mercaptan sulfur. Organic sulfur compounds such as sulfides, disulfides, and thiophene do not interfere. Elemental sulfur in amounts less than 0.0005 per cent by weight does not interfere. Hydrogen sulfide will interfere if not removed as described in this procedure.

NOTE 1.—An instrumental method, ASTM D 1323, Mercaptan Sulfur in Aviation Turbine Fuels (Amperometric-Potentiometric Method) gives results within the precision limits of this method, except Method D 1219 gives slightly higher results in the range of 0.0003 to 0.001 per cent mercaptan sulfur.

Section 4.—Add present Paragraphs 3(*a*) and 3(*b*), renumbering Paragraphs 4(*a*) to 4(*h*) as 3(*c*) to 3(*j*), respectively. Renumber this as Section 3; delete the heading "Purity of Reagents."

Renumber Sections 5, 6, and 7 as 4, 5, and 6.

Present Section 7.—Renumber as Section 6, and change to read:

6. The following criteria should be used for judging the acceptability of results (95 per cent probability):

(*a*) *Repeatability.*—Duplicate results by the same operator should not be considered suspect unless they differ by more than the following amounts:

Mercaptan Sulfur Content, per cent by weight	Repeatability, per cent by weight
0.0003 to 0.001	0.0001
0.0010 to 0.005	0.0002
0.0050 to 0.010	0.0005

(*b*) *Reproducibility.*—The result submitted by each of two laboratories should not be considered suspect unless the two results differ by more than the following amounts:

Mercaptan Sulfur Content, per cent by weight	Reproducibility, per cent by weight
0.0003 to 0.001	0.0004
0.0010 to 0.005	0.0006
0.0050 to 0.010	0.0009

D 1269 – 58,[3] Standard Method of Test for Tetraethyllead in Gasoline (Polarographic Method):

Revise as appended hereto.[17]

D 1323 – 56,[3] Standard Method of Test for Mercaptan Sulfur in Aviation Turbine Fuels (Amperometric Method):

Revise as appended hereto.[17]

Tentatives Continued Without Revision

The committee has reviewed the following tentatives which have stood two or more years without revision, and for good and sufficient reasons, discussed within the committee, recommends their continuation as tentatives without revision:

- **D 156 – 53 T,** Saybolt Color of Petroleum Products (Saybolt Chromometer Method)
- **D 240 – 57 T,** Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter
- **D 447 – 59 T,** Distillation of Plant Spray Oils
- **D 482 – 59 T,** Ash from Petroleum Oils
- **D 611 – 55 T,** Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents
- **D 721 – 56 T,** Oil Content of Petroleum Waxes
- **D 808 – 57 T,** Chlorine in New and Used Petroleum Products (Bomb Method)
- **D 873 – 57 T,** Oxidation Stability of Aviation Fuels (Potential Residue Method)
- **D 874 – 59 T,** Sulfated Ash from Lubricating Oils and Additives
- **D 910 – 57 T,** Specifications for Aviation Gasolines
- **D 1018 – 55 T,** Hydrogen in Petroleum Fractions
- **D 1022 – 59 T,** Peroxide Content of Light Hydrocarbons
- **D 1085 – 57 T,** Gaging Petroleum and Petroleum Products
- **D 1158 – 59 T,** Bromine Number of Petroleum Distillates by Color-Indicator Titration
- **D 1220 – 59 T,** Calibrating Upright Tanks
- **D 1264 – 53 T,** Water Washout Characteristics of Lubricating Greases
- **D 1266 – 59 T,** Sulfur in Petroleum Products and Liquefied Petroleum Gas (Lamp Method)
- **D 1317 – 57 T,** Chlorine in New and Used Lubricants (Sodium Alcoholate Method)
- **D 1318 – 59 T,** Sodium in Residual Fuel Oil (Flame Photometric Method)
- **D 1322 – 59 T,** Smoke Point of Aviation Turbine Fuels
- **D 1368 – 58 T,** Tetraethyllead in Trace Concentrations in Primary Reference Fuels
- **D 1465 – 57 T,** Blocking Point of Paraffin Wax
- **D 1477 – 57 T,** Freezing Point of Aviation Fuels
- **D 1478 – 57 T,** Low-Temperature Torque of Ball Bearing Greases
- **D 1479 – 57 T,** Emulsion Stability of Soluble Cutting Oils
- **D 1480 – 57 T,** Density and Specific Gravity of Viscous Materials by Bingham Pycnometer
- **D 1481 – 57 T,** Density and Specific Gravity of Viscous Materials by Lipkin Bicapillary Pycnometer
- **D 1500 – 58 T,** ASTM Color of Petroleum Products (ASTM Color Scale)
- **D 1548 – 59 T,** Vanadium in Navy Special Fuel Oil
- **D 1555 – 58 T,** Calculation of Volume and Weight of Benzene, Toluene, and Paraxylene
- **D 1657 – 59 T,** Specific Gravity of Light Hydrocarbons by Pressure Hydrometer
- **D 1658 – 59 T,** Carbon Number Distribution of Aromatic Compounds in Naphthas by Mass Spectrometry
- **D 1659 – 59 T,** Maximum Fluidity Temperature of Residual Fuel Oil
- **D 1661 – 59 T,** Thermal Stability of U. S. Navy Special Fuel Oil
- **D 1747 – 60 T,** Refractive Index of Viscous Materials

Standards Continued Without Revision

The committee recommends that the following standards which have stood six or more years without revision be continued as standards without revision as they are being used and, in the opinion of the committee, represent good practice:

- **D 287 – 55,** API Gravity of Petroleum Products, Hydrometer Method
- **D 446 – 53,** Conversion of Kinematic Viscosity to Saybolt Universal Viscosity
- **D 484 – 52,** Specifications for Stoddard Solvent
- **D 525 – 55,** Oxidation Stability of Gasoline (Induction Period Method)
- **D 565 – 45,** Carbonizable Substances in White Mineral Oil (Liquid Petrolatum)
- **D 567 – 53,** Calculating Viscosity Index
- **D 936 – 55,** Aromatic Hydrocarbons in Olefin-Free Gasolines by Silica Gel Adsorption
- **D 941 – 55,** Density and Specific Gravity of Liquids by Lipkin Bicapillary Pycnometer
- **D 943 – 54,** Oxidation Characteristics of Inhibited Steam-Turbine Oils
- **D 1015 – 55,** Freezing Points of High-Purity Hydrocarbons
- **D 1016 – 55,** Purity of Hydrocarbons from Freezing Points
- **D 1088 – 53,** Boiling Point Range of Polymerization-Grade Butadiene
- **D 1096 – 54,** 1-3-Butadiene in C_4 Hydrocarbon Mixtures by Ultraviolet Spectrophotometry
- **D 1215 – 54,** Definition and Specifications for Farm Tractor Fuels

D 1217 – 54, Density and Specific Gravity of Liquids by Bingham Pycnometer
D 1261 – 55, Effect of Grease on Copper
D 1262 – 55, Lead in New and Used Greases
D 1265 – 55, Sampling Liquefied Petroleum Gases
D 1267 – 55, Vapor Pressure of Liquefied Petroleum Gases
D 1268 – 55, Unsaturated Light Hydrocarbons (Silver-Mercuric Nitrate Method)
D 1298 – 55, Specific Gravity of Petroleum Liquids, Hydrometer Method

AMERICAN STANDARDS

The committee recommends the following for action by ASA Sectional Committee Z11 on Petroleum Products and Lubricants:

Reapproval of American Standards Revised in This Report:

Z11.24—1956, Flash Point by Tag Closed Tester (D 56)
Z11.10—1960, Distillation of Petroleum Products (D 86)
Z11.30—1952, Precipitation Number of Lubricating Oils (D 91)
Z11.9—1956, Water in Petroleum Product (D 95)
Z11.16—1960, Analysis of Lubricating Grease (D 128)
Z11.25—1958, Conradson Carbon Residue of Petroleum Products (D 189)
Z11.28—1957, Terms Relating to Petroleum (D 288)
Z11.26—1960, Distillation of Gas Oil and Similar Distillate Fuel Oils (D 158)
Z11.36—1958, Existent Gum in Fuels by Jet Evaporation (D 381)
Z11.37—1960, Knock Characteristics of Motor Fuels Below 100 Octane Number by the Motor Method (D 357)
Z11.107—1960, Kinematic Viscosity (D 445)
Z11.41—1960, Unsulfonated Residue of Petroleum Plant Spray Oils (D 483)
Z11.48—1953, Lead Antiknock Compounds in Gasoline (D 526)
Z11.69—1960, Knock Characteristics of Motor Fuels Below 100 Octane Number by the Research Method (D 908)
Z11.74—1960, Acetylenes in Butadiene, Butadiene Concentrates, and Butane-Butylene Mixtures (Silver Nitrate Method) (D 1020)
Z11.98—1958, Tetraethyllead in Gasoline (Polarographic Method) (D 1269)
Z11.105—1960, Mercaptan Sulfur in Aviation-Turbine Fuels (Ampero-Metric-Potentiometric Method) (D 1323)

Approval as American Standards:

D 129, Sulfur in Petroleum Products (Bomb Method)
D 1159, Bromine Number of Petroleum Distillates by Electrometric Titration
D 1160, Distillation at Reduced Pressure of Petroleum Products
D 1218, Refractive Index and Refractive Dispersion of Hydrocarbon Liquids
D 1219, Mercaptan Sulfur in Aviation Turbine Fuels (Color-Indicator Method)
D 1796, Water and Sediment in Fuel Oils by Centrifuge

American Standards Withdrawn:

Z11.94—1957, Analysis of 60 Octane Number Reference Fuel Blends (D 1095)

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[18]

This report has been submitted to letter ballot of the committee, which consists of 147 voting members; 86 members returned their ballots, of whom 76 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. M. SMITH,
Chairman.

W. T. GUNN,
Secretary.

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[18] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE D-3
ON
GASEOUS FUELS*

Committee D-3 on Gaseous Fuels held one meeting during the year: on Oct. 9, 1960, in Atlantic City, N. J.

The committee consists of 33 members, of whom 14 are classified as producers, 7 as consumers, and 12 as general interest members.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1960 Annual Meeting, Committee D-3 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method of Test for:

Calorific Value of Gases in the Natural Gas Range by the Continuous Recording Calorimeter (D 1826 - 61 T).

Revision of Tentative Method for Analysis of:

Carbureted Water Gas by the Mass Spectrometer (D 1302 - 53 T).

These recommendations were accepted by the Standards Committee on February 15, 1961, and both the new and revised methods will appear in the 1961 Book of ASTM Standards, Part 8.

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Standard Methods of:

Test for Calorific Value of Gaseous Fuels by the Water-Flow Calorimeter (**D 900 - 55**),

Measurement of Gaseous Fuel Samples (**D 1071 - 55**),

Analysis of Natural Gases by the Volumetric-Chemical Method (**D 1136 - 53**),

Sampling Natural Gas (**D 1145 - 53**), and

Sampling Manufactured Gas (**D 1247 - 54**).

The committee recommends the reapproval of the following standard with the editorial change as indicated:

Standard Method of Sampling Liquefied Petroleum Gases (D 1265 - 55):

New Note.—Add a new note to read as follows:

NOTE.—Samples to be tested for presence of corrosive compounds or sulfur compounds should be taken in stainless steel containers equipped with stainless steel valves; otherwise, determination of mercaptans and hydrogen sulfide, for example, may be misleading.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee III on Determination of Calorific Value of Gaseous Fuels (A. F. Cascioli, chairman) completed a tentative method for heating value determination by the recording calorimeter (D 1826) which covers natural gases in the range from 900 to 1200 Btu. Consideration is now being given to extending this range by lowering the minimum heating value to 800 Btu.

Subcommittee IV on Determination of Specific Gravity and Density of Gaseous Fuels (E. E. Stovall, chairman) is studying new developments in methods of specific gravity determination in connection with revision of the present method. Material is expected to be available for review at a meeting later in the year.

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* Sixty-fourth Annual Meeting of the Society. June 25–30, 1961.

Subcommittee V on Determination of Special Constituents of Gaseous Fuels (O. W. Lusby, chairman) met on Oct. 9, 1960, and discussed procedures for determination of total sulfur and hydrogen sulfide in small concentrations of less than one grain per 100 cu ft. No ASTM methods for their determination in such quantities are now available. It was felt that the methylene blue method for hydrogen sulfide could be developed at a moderate expenditure and that availability of a method along these lines was desirable. An estimate of the cost of its development was made and presented to interested industrial groups. However, sufficient industry interest appeared to be lacking as no funds were made available.

Subcommittee VI on Determination of Water Vapor Content of Gaseous Fuels (E. G. Hammerschmidt, chairman) submitted a proposal for a research project for pipe line research to develop a water vapor recorder capable of accurate results under all field and plant operating conditions. However, the project is not considered to be of industry-wide interest requiring immediate action, and approval has not been accorded it.

The subcommittee is studying the possibility of extending the scope of the present dew-point method for determination of water vapor to include determination of dew points other than of water. This includes a canvass of several companies now using an auxiliary magnifying device for the purpose and its test by subcommittee members.

Subcommittee VII on Complete Analysis of Chemical Composition of Gaseous Fuels (D. V. Kniebes, chairman) revised the Tentative Method of Analysis of Carbureted Water Gas by the Mass Spectrometer (D 1302 – 53 T). Revision of a corresponding method for natural gas analysis is now under way.

A proposed draft of a tentative method for natural gas analysis by gas chromatography, previously distributed to members, was discussed at the June 27 meeting. Several suggestions were offered, particularly the desirability of covering C_6 and higher fractions. A revised draft is expected to be in readiness for early committee distribution.

Liaison is being maintained with Committee D-2 on Petroleum Products and Lubricants on development of a method for analysis of liquefied petroleum gases by gas chromatography.

This report has been submitted to letter ballot of the committee, which consists of 33 members; 27 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

D. V. KNIEBES,
Chairman.

K. R. KNAPP,
Secretary.

REPORT OF COMMITTEE D-4
ON
ROAD AND PAVING MATERIALS*

Committee D-4 on Road and Paving Materials held two meetings during the year: in Atlantic City, N. J. on July 1, 1960, and in Cincinnati, Ohio, on February 1, 1961.

Fred Hubbard was elected as an Honorary Member by unanimous vote of Committee D-4.

The committee lost four members by death: P. J. Freeman, P. D. Miesenhelder, H. F. Clemmer, and Carl Bussow. In recognition of the many valuable contributions of these members to the work of the committee and of the loss which members felt in their passing, appropriate memorial resolutions were prepared and adopted and are being entered on the permanent records of the committee and the Society.

Subcommittee B-4 on Ductility was deactivated on February 1, 1961, and its duties and members were transferred to Subcommittee B-24 which was renamed "Penetration and Ductility Tests."

Subcommittee C-14 on Standard Sizes of Coarse Aggregates for Highway Construction was also deactivated and its members and specification responsibility were assigned to Subcommittee B-9 on Size of Aggregates.

A. B. Cornthwaite announced his resignation as chairman of Committee D-4 at the Feb. 1, 1961, meeting. In accordance with regulations governing Committee D-4, R. E. Bollen was appointed to fill the post of chairman for the remainder of the current term of office. W. H. Goetz was appointed to replace Mr. Bollen as first vice-chairman.

A Symposium on Microviscometry was arranged for the 1961 Annual Meeting. Arrangements were also made for presentation of several papers at the session on Road and Paving Materials at the 1961 Annual Meeting.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods and specifications as appended hereto:[1]

Tentative Methods of:

Test for Recovery of Asphalt from Solution by Abson Method,
Testing Concrete Joint Sealers, Cold-Application Type,
Testing Jet-Fuel-Resistant Joint Sealer for Concrete, Cold-Application Elastic Type, and
Testing Jet-Fuel-Resistant Concrete Joint Sealer, Hot-Poured Elastic Type.

Tentative Specifications for:

Concrete Joint Sealer, Cold-Application Type
Jet-Fuel-Resistant Concrete Joint Sealer, Cold Application Elastic Type, and
Jet-Fuel-Resistant Concrete Joint Sealer, Hot Poured Elastic Type.

REVISIONS OF TENTATIVE

The committee recommends the revision as follows of the Tentative Specification for Crushed Stone, Crushed Slag, specific gravity determination in connection with revision of the present method. Material is expected to be available for review at a meeting later in the year.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] The new tentatives appear in the 1961 Book of ASTM Standards, Part 4.

Subcommittee V on Determination of Special Constituents of Gaseous Fuels (O. W. Lusby, chairman) met on Oct. 9, 1960, and discussed procedures for determination of total sulfur and hydrogen sulfide in small concentrations of less than one grain per 100 cu ft. No ASTM methods for their determination in such quantities are now available. It was felt that the methylene blue method for hydrogen sulfide could be developed at a moderate expenditure and that availability of a method along these lines was desirable. An estimate of the cost of its development was made and presented to interested industrial groups. However, sufficient industry interest appeared to be lacking as no funds were made available.

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The subcommittee is studying the possibility of extending the scope of the present dew-point method for determination of water vapor to include determination of dew points other than of water. This includes a canvass of several companies now using an auxiliary magnifying device for the purpose and its test by subcommittee members.

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Liaison is being maintained with Committee D-2 on Petroleum Products and Lubricants on development of a method for analysis of liquefied petroleum gases by gas chromatography.

This report has been submitted to letter ballot of the committee, which consists of 33 members; 27 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

D. V. KNIEBES,
Chairman.

K. R. KNAPP,
Secretary.

REPORT OF COMMITTEE D-4

ON

ROAD AND PAVING MATERIALS*

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NEW TENTATIVES

The committee recommends for publication as tentative the following methods and specifications as appended hereto:[1]

Tentative Methods of:

Test for Recovery of Asphalt from Solution by Abson Method,
Testing Concrete Joint Sealers, Cold-Application Type,
Testing Jet-Fuel-Resistant Joint Sealer for Concrete, Cold-Application Elastic Type, and
Testing Jet-Fuel-Resistant Concrete Joint Sealer, Hot-Poured Elastic Type.

Tentative Specifications for:

Concrete Joint Sealer, Cold-Application Type
Jet-Fuel-Resistant Concrete Joint Sealer, Cold Application Elastic Type, and
Jet-Fuel-Resistant Concrete Joint Sealer, Hot Poured Elastic Type.

REVISIONS OF TENTATIVE

The committee recommends the revision as follows of the Tentative Specification for Crushed Stone, Crushed Slag, and Gravel for Single or Multiple Bituminous Surface Treatments (**D 1139 - 57 T**):[2]

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] The new tentatives appear in the 1961 Book of ASTM Standards, Part 4.

Table I.—Change the maximum percentage by weight of total sample for aggregate sizes 5 and 6 from "5.0" to "10.0." After the words "Flat or elongated pieces,[b]" add the words "retained on the $\frac{3}{8}$-in. sieve."

TENTATIVE REVISION OF STANDARD

The committee recommends the tentative revision as appended hereto[3] of the Standard Methods of Testing Emulsified Asphalts (**D 244 - 60**).[2]

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends revisions as indicated of the following standards and their reversion to tentative:

Standard Methods of Test for:

Amount of Materials Finer Than No. 200 Sieve in Aggregate (**C 117 - 49**).[2] Revise as appended hereto.[4]

Sieve Analysis of Fine and Coarse Aggregates (**C 136 - 46**)[2] (Jointly with Committee C-9). Revise as appended hereto.[4]

Standard Specifications for:

Bituminous Mixing Plant Requirements (**D 995 - 55**).[2] Revise as appended hereto.[4]

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods of Test for:

Vacuum Distillation of Liquid and Semi-Solid Asphaltic Materials to Obtain a Residue of Specified Penetration (**D 1189 - 52 T**),[2] and

Engler Specific Viscosity of Tar Products (**D 1665 - 59 T**).[5]

ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that the Tentative Method of Test for Penetration of Bituminous Materials (**D 5 - 59 T**)[5] be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated.

New Section 9.—Add a new Section 9 entitled "Precision" to read as follows:

> 9. The following criteria should be used for judging the acceptability of results (95 per cent probability) for penetration tests at 77 F, 100 g, 5 sec:
>
> (*a*) *Repeatability.*—Duplicate results by the same operator should not be considered suspect unless they differ by more than 4 per cent.
>
> (*b*) *Reproducibility.*—The results submitted by each of two laboratories should not be considered suspect unless the two results differ by more than 10 per cent.

WITHDRAWAL OF STANDARD

The committee recommends the withdrawal of the Standard Method of Test for Penetration of Bituminous Materials (**D 5 - 52**).[2]

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following 23 standards which have stood for six or more years without revision:

Standard Methods of Test for:

Resistance to Plastic Flow of Fine-Aggregate Bituminous Mixtures by Means of the Hubbard-Field Apparatus (**D 1138 - 52**),

Distillation of Cutback Asphaltic Products (**D 402 - 55**),

Softening Point of Bituminous Materials (Ring-and-Ball Method) (**D 36 - 26**),

Softening Point of Tar Products (Cube-in-Water Method) (**D 61 - 38**),

Hot Extraction of Asphaltic Materials and Recovery of Bitumen by the Modified Abson Procedure (**D 762 - 49**),

Float Test for Bituminous Materials (**D 139 - 49**),

Sieve Analysis of Mineral Filler (**D 546 - 55**),

Abrasion of Coarse Aggregate by Use of Los Angeles Machine (**C 131 - 55**),

Abrasion of Rock by Use of the Deval Machine (**D 2 - 33**),

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[2] 1958 Book of ASTM Standards, Part 4.

[3] The tentative revision appears in the 1961 Book of ASTM Standards, Part 4.

[4] The revised methods and specification appear in the 1961 Book of ASTM Standards, Part 4.

[5] 1959 Supplement to Book of ASTM Standards, Part 4.

Toughness of Rock (**D 3 - 18**),
Abrasion of Graded Coarse Aggregate by Use of the Deval Machine (**D 289 - 55**),
Sampling Bituminous Materials (**D 140 - 55**),
Sampling Bituminous Paving Mixtures (**D 979 - 51**),
Ductility of Bituminous Materials (**D 113 - 44**),
Effect of Water on Cohesion of Compacted Bituminous Mixtures (**D 1075 - 54**),
Sulfonation Index of Road Tars (**D 872 - 48**),
Preformed Expansion Joint Fillers for Concrete (Nonextruding and Resilient Type) (**D 545 - 49**),
Residue of Specified Penetration (**D 243 - 36**),
Volume Correction Table for Tar and Coal-Tar Pitch (**D 633 - 44**),
Specific Gravity of Road Oils, Road Tars, Asphalt Cements, and Soft Tar Pitches (**D 70 - 52**), and
Specific Gravity of Asphalts and Tar Pitches Sufficiently Solid to be Handled in Fragments (**D 71 - 52**).

Specifications for:

Tar (**D 490 - 47**), and
Crushed Stone and Crushed Slag for Bituminous Macadam Base and Surface Courses of Pavements (**D 693 - 54**).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Methods of Test for:

Resistance to Deformation and Cohesion of Bituminous Mixtures by Means of Hveem Apparatus (**D 1560 - 58 T**),
Preparation of Test Specimens of Bituminous Mixtures by Means of California Kneading Compactor (**D 1561 - 58 T**),
Scratch Hardness of Coarse Aggregate Particles (**C 235 - 57 T**),
Loss on Heating of Oil and Asphaltic Compounds (**D 6 - 39 T**),
Coating and Stripping of Bitumen-Aggregate Mixtures (**D 1664 - 59 T**), and
Concrete Joint Sealers (**D 1191 - 52 T**).

Tentative Specifications for:

Asphalt Cement for Use in Pavement Construction (**D 946 - 47 T**),
Crushed Stone, Crushed Slag, and Crushed Gravel for Dry-Bound or Water-Bound Macadam Base Courses (**D 694 - 57 T**),
Mineral Filler for Bituminous Paving Mixtures (**D 242 - 57 T**),
Coarse Aggregate for Bituminous Paving Mixtures (**D 692 - 59 T**),
Hot-Mixed, Hot-Laid Asphalt Paving Mixtures (**D 1663 - 59 T**), and
Concrete Joint Sealer, Hot-Poured Elastic Type (**D 1190 - 52 T**).

TENTATIVE REVISIONS CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the tentative revisions as indicated of the following standards:

Standard Methods of Testing:

Emulsified Asphalts (**D 244 - 60**), tentative revision issued June, 1958.
Preformed Expansion Joint Fillers for Concrete (Nonextruding and Resilient Types) (**D 545 - 49**), tentative revision issued June, 1956.

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following standards:

Standard Specifications for Cut-back Asphalt (Rapid-Curing Type) (D 597 - 46),[2] and

Standard Specifications for Cut-back Asphalt (Medium-Curing Type). (D 598 - 46):[2]

Sections 3(c).—Add to the end of this paragraph in both specifications: "shall be used except that the distillate fractions shall be reported on the basis of percentage by volume of the total distillate to 680 F."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[6]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee A-4 on Papers (C. L. Monismith, chairman) sponsored six papers at a highly successful technical session at the 1960 Annual Meeting. Arrangements have been made for the presentation of five papers at a Symposium on Microviscometry to be held at the 1961 Annual Meeting. Arrangements have also been made for presentation of several papers at the Technical Session on Road and Paving Materials at the 1961 Annual Meeting.

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[6] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee A-5 on Finance (P. F. Phelan, chairman) was appointed in February 1960 with the responsibility of developing a suitable program for financing the necessary activities of Committee D-4.

Subcommittee B-1 on Asphalt Content (H. F. Waller, chairman) has recommended that Method D 1189 be adopted as standard. The subcommittee is undertaking a study of the need for test methods for obtaining residues for test for elastomer modified asphaltic cutback.

Subcommittee B-2 on Physical Tests for Compressed Bituminous Mixtures (L. F. Rader, chairman) has task forces actively studying methods under their jurisdiction. Task Force I and IV have made various recommendations for changes in Method D 1559. Task Force V is studying and preparing recommendations concerning standardization of kneading compactors for possible revisions of Method D 1561.

Subcommittee B-5 on Softening Point (D. F. Fink, chairman) has been studying means for improving the precision of Method D 36. As a result of a cooperative test program a report entitled "A Statistical Comparison of a Modified Softening Point Method with ASTM D 36 - 26, Softening Point of Bituminous Materials, Ring and Ball Method" is being recommended for publication in *Materials Research & Standards.*

Subcommittee B-6 on Extraction and Recovery of Constituents from Bituminous Mixtures (E. W. Klinger, chairman) has recommended the adoption of the Tentative Method of Test for Recovery of Asphalt from Solution by the Abson Method. The subcommittee is also working on preparation of three tentative extraction test methods and expects to complete this work before the 1961 Annual Meeting.

Subcommittee B-7 on Viscosity and Float Test (C. A. Benning, chairman) has been concerned with the float test and the development of the Engler viscosity method for road tars. This subcommittee has been directed to investigate other viscosity test methods, particularly those of the capillary tube type.

Subcommittee B-9 on Size of Aggregates (Fred Hubbard, chairman) has reverted Methods C 117 and C 136 to tentative to incorporate major changes. The subcommittee has taken over the responsibilities of recently deactivated Subcommittee C-14 on Standard Sizes of Coarse Aggregates for Highway Construction.

Subcommittee B-12 on Structural Properties of Mineral Aggregates (Stanton Walker, chairman) has recommended publication in *Materials Research & Standards* of a complete report on the study of the Los Angeles test by D. O. Woolf's special committee. A cooperative investigation of a crushing test for coarse aggregate, developed in Great Britain, has been considered.

Subcommittee B-14 on Specific Gravity (H. T. Williams, chairman) has planned a collaborative investigation of the "Rice" method of test for maximum specific gravity of uncompacted bituminous paving mixtures. A task force of four members has been appointed to prepare a program and six laboratories have volunteered to participate.

Subcommittee B-16 on Setting Qualities of Bituminous Materials (A. B. Cornthwaite, chairman) has circulated to

Committee D-4, for information, a Method of Test for Curing Rate of Cutback Asphalts as a result of cooperative tests and plans to submit this method to letter ballot as soon as all test data can be summarized and evaluated.

Subcommittee B-17 on Emulsion Tests (P. E. McCoy, chairman) recommended adoption of a method of test for coating and water resistance of coarse aggregate mixing emulsions as a tentative revision of Method D 244. The subcommittee has appointed a task force to consider and develop cationic emulsion tests.

Subcommittee B-19 on Accelerated Tests for Durability of Bituminous Materials (D. C. Taylor, chairman) has a task force working on a method of test for aging index of bituminous materials. A cooperative testing program is also under way on the thin film oven test.

Subcommittee B-24 on Penetration Test (J. Y. Welborn, chairman) undertook a series of cooperative tests to establish the precision of Method D 5 – 59 T. They have recommended withdrawal of Method D 5 – 52 and the adoption as standard of Method D 5 – 59 T with inclusion of a precision statement. The name of the committee was changed to "Penetration and Ductility Tests" and has taken over the responsibilities of the recently deactivated Subcommittee B-4 on Ductility.

Subcommittee B-26 on Effect of Water on Bituminous Coated Aggregates (W. H. Goetz, chairman) has five active task forces. Consideration has been given to the formulation of a precision statement for Method D 1075. The subcommittee is also working on preparation of a tentative method of test for stripping of bitumen which would provide for a measure of film retention by means of the lithium tracer salt technique.

Subcommittee C-1 on Asphalt Cements (A. R. Curtis, chairman).—Subcommittee letter ballots are being taken on changing the number of penetration grades in Specification D 946 to five, on substituting the thin film oven test for the loss on heating test, and on eliminating the test limits for per cent ash.

Subcommittee C-4 on Emulsified Asphalts (C. E. Proudley, chairman) has a task force working on specifications for cationic emulsions and a task force considering the revision of the format of specifications for asphalt emulsions.

Subcommittee C-8 on Water-Bound Roads (J. E. Wood, chairman) has established a task force to explore the testing of fine materials derived from crushed stone, slag, or natural sources since Specifications D 694 have been continued as tentative due to inadequacies in Section (*b*) of that specification.

Subcommittee C-12 on Bituminous Surface Treatments (J. O. Izatt, chairman) recommended a revision to Specification D 1139 based on a survey of limits for flat and elongated pieces for certain aggregate sizes.

Subcommittee C-13 on Bituminous Paving Plant Operations (J. Ward, chairman) prepared extensive revisions to Specification D 995 and recommended the specification be reverted to tentative.

Subcommittee D-1 on Calcium and Sodium Chloride for Use as Road Materials (J. D. Lindsay, chairman) is endeavoring to prepare a method of test for determining moisture in sodium chloride and calcium chloride.

Subcommittee D-2 on Highway Traffic Marking Materials (Paints, Insets and Strips) (N. G. Smith, chairman) is considering the type of specification needed for traffic paints and plans appointment of task forces for this purpose.

Subcommittee D-3, Joint Fillers and

Asphalt Planks (E. R. Oglio, chairman) prepared the three tentative specifications for concrete joint sealers, and three tentative methods of test for concrete joint sealers.

This report has been submitted to letter ballot of the committee, which consists of 186 members; 85 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. E. BOLLEN,
Chairman.

B. F. KALLAS,
General Secretary.

REPORT OF COMMITTEE D-5
ON
COAL AND COKE*

Committee D-5 on Coal and Coke held one meeting during the year in Atlantic City, N. J., on June 29, 1960. Three meetings of the Advisory Subcommittee were held in Atlantic City, N. J., on June 28, 1960; in Pittsburgh, Pa., September 27, 1960; and in Cincinnati, Ohio, January 30, 1961. Subcommittees and task groups also held meetings in Atlantic City, in Pittsburgh, and in Cincinnati.

During the year the committee lost two members due to retirement and three members were added. The 67 voting members are classified as: producer, 19; consumers, 27; and general interest members, 21. The present committee consists of the Advisory and 11 technical subcommittees having 15 sections and task groups. Included in these groups are 29 consulting members who are specialists in sampling, testing, analysis, classification, and related activities.

The retiring chairman of this committee, O. W. Rees, was honored by the Society at the 1960 Annual Meeting with the Society's Award of Merit.

RECOMMENDATION ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1960 Annual Meeting, Committee D-5 presented to the Society through the Administrative Committee on Standards the recommendation that the Method of Test for Plastic Properties of Coal by the Gieseler Plastometer be published as tentative. This recommendation was accepted by the Standards Committee on September 29, 1960, and the method appears in the 1960 Supplement to Book of ASTM Standards, Part 8, bearing the designation D 1812 - 60 T.

NEW TENTATIVE

The committee recommends for publication as tentative the Proposed Tentative Method of Test for Fusibility of Coal Ash as appended hereto.[1]

TENTATIVE REVISION OF STANDARD

The committee recommends tentative revisions as follows of the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (**D 271 - 58**):[2]

Section 18(b).—Change to read as follows:

(*b*). *Volatile Matter By Modified Procedure.*—All fuels that spark when the volatile matter is determined by the methods described in Sections 17 and 19 shall be treated as follows: The sample shall be given a preliminary gradual heating such that a temperature of 600 ± 50 C is reached in 6 min (Notes 12, 13, 14). After this preliminary heating the sample is heated for exactly 6 min at 950 ± 20 C. If sparking is then observed the determination shall be rejected and the test repeated until no sparking occurs either during the preliminary heating or during the 6-min period at 950 C. Remove crucible from furnace and cool on a metal cooling block and weigh (Note 15). The percentage loss in weight minus the per cent moisture is the volatile matter. All analyses by this method shall be marked to indicate that the modified procedure was used.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] The new tentative appears in the 1961 Book of ASTM Standards, Part 8.

[2] 1958 Book of ASTM Standards, Part 8.

New Notes.—Add the following four new notes following Section 18(*b*), renumbering subsequent notes accordingly:

NOTE 12.—If a tubular furnace of the Fieldner type is used for the determination of volatile matter, the preliminary gradual heating may be accomplished by moving the crucible to predetermined positions in the cooler top zone of the furnace. Due to variations in the heating characteristics of the furnace, the operator must determine by the thermocouple method in Section 19 for each furnace the proper position to meet preliminary heating rate as specified. It is also possible to use a mechanical device to lower the crucible into the furnace.

NOTE 13.—If electric muffle furnaces are used the heating may be accomplished by using two furnaces situated side by side, one furnace being controlled at 550 ± 10 C, the other at 950 ± 10 C. The crucible containing the sample is placed on a nichrome support $\frac{3}{8}$ to $\frac{1}{2}$ in. high, and placed in the muffle furnace controlled at 550 ± 10 C for exactly 6 min, after which time it is rapidly transferred along with its nichrome support to the second muffle furnace, controlled at 950 ± 10 C, and allowed to remain for exactly 6 min.

NOTE 14.—If the Meker burner method described in Paragraph (*c*) is used, the rate of heating specified in (*b*) shall be observed.

NOTE 15.—To insure uniformity of results the cooling period should be kept constant and should not be prolonged beyond 15 min.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Method of Test for Equilibrium Moisture of Coal at 96 to 97 per cent Relative Humidity and 30 C (**D 1412 - 56 T**)[2] be approved for reference to letter ballot of the Society for adoption as standard without revision.

REAPPROVAL OF STANDARDS

All the standards under the jurisdiction of this committee that have stood for six years or more without revision have been reviewed and are still considered to be in accord with present practice. The Committee recommends the reapproval of these standards without revision.

TENTATIVE REVISION CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the tentative revision of Standard Definitions of Terms Relating to Coal and Coke (**D 121 - 30**).[2]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Advisory Subcommittee (R. L. Coryell, chairman) approved the formation of a new Subcommittee XXV on Physical Tests of Briquets. It also approved the reactivation of Subcommittee XVIII on Classification of Coal. Task Group D was approved to coordinate the subcommittee programs for the preparation and revision of methods of sampling and analysis of coal for moisture. This group under the chairmanship of W. W. Anderson made several helpful recommendations which have been transmitted to Subcommittees XXI and XXIII. New chairmen were appointed for Subcommittees VII, XV, XVIII, XXI, and XXIV. Work has been started by a special task group headed by the committee Vice Chairman, W. M. Bertholf, on the review of the scopes of the subcommittees.

Subcommittee II on Nomenclature and

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Definitions (C. H. Sawyer, chairman) has been actively engaged in reviewing the new tentatives on coal and coke for consistency of terminology and also in considering terms referred by other subcommittees. Particular attention has been given to calorimetry terms to be used in the proposed method utilizing the adiabatic calorimeter prepared by Task Group C, as reported later.

Subcommittee VII on Physical Characteristics of Coal (A. Bogot, chairman) has continued work on revision of the Method of Test for Grindability of Coal by the Hardgrove-Machine Method (D 409 – 51). The previous chairman, H. F. Yancey, who served effectively on this subcommittee for many years, has retired from the U. S. Bureau of Mines. With the greatly enlarged scope, this subcommittee is reviewing eight other standards including tests for screen (sieve) analysis of coal.

Subcommittee XV on Plasticity and Swelling of Coal (C. R. Montgomery, chairman) is considering new procedures for and possible revision of Method of Test for Free-Swelling Index of Coal (D 720 – 57). Task groups are preparing methods of tests for expansion properties of coal by the movable-wall oven and the sole-heated oven. Consideration is being given to use of different operating procedures for the 12-in. and the 18-in. test ovens. Investigations are being made of possible improvements in the new Method D 1812 – 60 T mentioned earlier in this report and the constant torque modification of the Gieseler apparatus.

Subcommittee XVIII on Classification of Coal (R. A. Glenn, chairman) has been reactivated and 15 members appointed. At the first meeting in Cincinnati, plans were made for reviewing and possibly up-dating present Specifications D 388 – 38 and D 389 – 37. Task groups have been designated to implement the program.

Subcommittee XX on Sampling and Fineness of Pulverized Coal (E. L. Rogers, chairman) has not held any meetings during the year. Work is in progress on revision of Method D 197 – 30.

Subcommittee XXI on Methods of Analysis (J. B. McIlroy, chairman) has completed a tentative revision of Method D 271 – 58, consisting of a modified method for determination of volatile matter, as noted earlier in this report. Work is progressing on methods for determination of chlorine and forms of sulfur in coal. This subcommittee is now undertaking the tasks of (1) developing referee and non-referee methods for determination of total moisture in coal, and (2) considering modification of certain portions of present standards covering the determination of moisture.

Subcommittee XXII on Physical Tests of Coke (W. L. Glowacki, chairman) is considering possible revisions in standard Methods D 141 – 48, D 293 – 50, and D 294 – 50 covering shatter, sieve, and tumbler tests of coke. Other physical tests of coke will be similarly reviewed for possible revision. This subcommittee is particularly interested in the activities of Working Group 8 on Coke of ISO TC/27, and the chairman has attended two meetings of that group in Europe, as described later under Subcommittee XXVII.

Subcommittee XXIII on Sampling (W. M. Bertholf, chairman) has reached general agreement on the fundamentals involved in methods and equipment for mechanical sampling of coal and preparation of laboratory samples. It is hoped that final drafts of these methods will be approved by the subcommittee this year and submitted to the committee for action. Upon completion of the method for mechanical sampling, consideration will be given to hand sampling of coal. Matters pertaining to sampling of coke are being handled by the chairman until there is sufficient need for reactivating the coke sampling section.

Subcommittee XXIV on Statistics (W. M. Bertholf, chairman) has been organized as a service group (1) to establish uniform practices of obtaining and analyzing precision test data for coal and coke test methods; (2) to assist other subcommittees in establishing precision limits; (3) to review the precision statements in proposed methods or revisions thereof; and (4) to review and recommend desirable changes in subcommittee interlaboratory tests prior to experimentation.

At the first meeting in Cincinnati plans were proposed for active liaison with Committee E-11 on Quality Control of Materials. The chairman has unofficially been performing this service for the committee for several years and the formation of this subcommittee is intended to make his work official and to provide several well-qualified assistants.

Subcommittee XXV on Physical Tests of Briquets (chairman to be appointed). The formation of this subcommittee has been approved by the Advisory Subcommittee, and the scope and related matters are now being considered.

Subcommittee XXVII, American Group ISO/TC 27 on Solid Mineral Fuels (W. H. Ode, chairman) has held meetings concurrently with regular D-5 subcommittee meetings plus two additional meetings in Pittsburgh, Pa. The four Research Sections on (1) Sampling, (2) Preparation, (3) Coke Testing, and (4) Analysis have been most active and have reviewed the numerous pertinent ISO documents provided through the American Standards Assn.

ISO TC/27 Working Groups 7 and 8 on Sampling and Coke Testing held meetings in Madrid, Spain, November 9 to 16, 1960. W. L. Glowacki and A. A. Orning from this committee attended as American representatives.

A draft proposal for the determination of the gross calorific value of coal by the adiabatic bomb calorimeter, based on the work of Task Group C, has been prepared and submitted as the USA contribution to ISO TC/27 at their request. A small representation of Committee D-5 members is attending the Sixth Plenary Meeting of ISO TC/27, to be held in London the week of June 26, 1961.

Financing the participation of USA delegates to ISO meetings in Europe is becoming an increasingly serious problem to this committee. Fortunately this year these expenses were largely defrayed by the organizations of the representatives or by the representatives themselves, but there is no certainty that this will continue. It is necessary that the committee have on-the-spot representation at the ISO meetings for effective USA participation in the ISO programs. Contributions to the ASTM fund by interested organizations are essential for continued active participation in this international work.

Task Group C on the Adiabatic Calorimeter (A. O. Blatter, chairman) has completed the third draft of the method of test for gross calorific value of solid fuel by the adiabatic calorimeter and has received letter ballot approval of its members. Interlaboratory tests had been made in 15 laboratories on samples of four coals representing different areas, and data were obtained to establish calibration requirements and the precision limits between and within laboratories. This method is being recommended for approval by the committee as a proposed tentative method.

Task Group D on Moisture in Coal Determinations (W. W. Anderson, chairman) has reviewed the existing methods for preparation and analysis of coal samples for moisture, and has recommended specific assignments for Subcommittees XXI and XXIII. Task Group D will follow the moisture aspects of the work of these subcommittees and continue to make recommendation for the coordination of this work.

This report has been submitted to letter ballot of the committee, which consists of 67 voting members; 52 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. L. CORYELL,
Chairman.

R. F. ABERNETHY,
Secretary.

REPORT OF COMMITTEE D-6
ON
PAPER AND PAPER PRODUCTS*

Committee D-6 on Paper and Paper Products held two meetings during the year: on June 30, 1960, in Atlantic City, N. J., and on Feb. 23 and 24, 1961 in New York, N. Y. The Advisory Committee, subcommittees, and task groups also met at these times.

Committee D-6 consists of 101 members of whom 85 are voting members; 30 are classified as producers, 22 as consumers, and 49 as general interest members.

An operating procedure for the joint development of standard methods of testing paper and paper products by both ASTM and the Technical Association of the Pulp and Paper Industry has been developed. Upon approval by both Committee D-6 and TAPPI, joint subcommittees will be formed to implement this procedure.

The Joint TAPPI-ASTM Committee D-6 Symposium on Relative Humidity, Its Measurement and Control, held during the TAPPI Testing Conference in Grand Rapids, Mich., on September 28, 1960, was enthusiastically received. The purpose of the symposium was to focus attention on the need for accurate control of temperature and humidity, to point out the danger of relying on inadequate, inaccurate and insensitive measuring and controlling equipment and to present designs for enclosures wherein close control of temperature and humidity can be realized.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions of the following standards as indicated, and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

Standard Method of Test for Reducible Sulfur in Paper (D 984 - 57):[1]

New Section.—Add a new Section 2 on Significance to read as follows, renumbering subsequent sections accordingly.

2. *Significance.*—(*a*) Paper showing less than 0.0008 per cent reducible sulfur when tested by this method may be regarded as nontarnishing toward polished metals, as far as sulfur is concerned. However, this value is not intended to define non-tarnish paper *per se.*

(*b*) This method will satisfactorily determine reducible sulfur content as low as 0.0001 per cent (1 ppm) in paper if the blank determination is negative. In case the blank indicates a significant amount of reducible sulfur present in the reagents or atmosphere, sensitivity of the determination is correspondingly reduced.

Standard Method of Static Bending Test for Corrugated Paperboard (D 1098 - 52):[1]

New Section.—Add a new Section 2 on Significance to read as follows, renumbering subsequent sections accordingly:

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[1] 1958 Book of ASTM Standards, Part 6.

2. *Significance.*—The static bending test makes use of the relationship between load and deflection which may be used to determine the modulus of elasticity or stiffness of corrugated fiberboard. This stiffness is one factor in the evaluation of the strength of corrugated fiberboard. The deflection of a beam under load varies inversely as the modulus of elasticity, that is, the higher the modulus the less the deflection. This physical property of corrugated fiberboard is influenced by the kind, weight and thickness of the facings; the quality and thickness of the corrugating medium; the form of the corrugations; and the quality of the adhesive bond between the component paperboards of the built-up structural material.

While the results of static bending tests are indicative of the strength characteristics of corrugated board it must be recognized that when made up into containers such board seldom acts as a simple beam or column due to the effect of scores and other features of construction.

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six years or more without revision:

Standard Methods of Test for:

Machine Direction of Paper (**D 528 - 41**),
Printing Ink Permeation of Paper (Castor Oil Test) (**D 780 - 46**),
Absorption by Bibulous Papers of Water and Writing Ink (**D 824 - 47**),
Bleeding Resistance of Asphalted Paper at Elevated Temperature (**D 917 - 49**),
Blocking Resistance of Paper and Paperboard (**D 918 - 49**),
Crease Retention of Wrapping Paper (**D 920 - 49**),
Paraffin Wax Absorptiveness of Paper (**D 983 - 50**),
45-deg, 0-deg Directional Reflectance for Blue Light (Brightness) of Paper (**D 985 - 50**), and
Lint of Paper Towels (**D 1163 - 53**).

Standard Methods for:

Creasing Paper for Permeability Tests (**D 1027 - 51**).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentative:

Tentative Methods of Test for:

Contrast Gloss of Paper at 57.5 deg (**D 1222 - 52 T**).

EDITORIAL CHANGES

The committee recommends editorial changes as follows in the Standard Method of Test for Water-Soluble Acidity or Alkalinity (**D 548 - 41**):[1]

Section 7.—In line 5, change "sodium oxide (Na_2O)" to "sodium hydroxide (NaOH)." Change the second formula to read "NaOH per cent = $(T_2 \times N_2 + t \times N_1) \times 0.04 \times 100$."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Sampling and Conditioning (T. W. Lashof, chairman).—A task group is working on a recommended form for the precision statement for methods based on D 1749, Tentative Recommended Practice for Interlaboratory Evaluation of Test Methods. A new permanent task group has been formed to advise task groups of other subcommittees of the committee in the planning and analysis of round-robin tests. Two methods for conditioning are in process.

Subcommittee II on Chemical Methods (A. P. Tracy, chairman).—Methods for mold resistance and quantitative content of mineral fillers are being balloted upon. Methods D 276 and D 1030 are being studied with a view to combining them into a single method for fiber identification.

Subcommittee III on Paper Testing (L. Price, chairman) is balloting on thickness of paper and paper products, stretch of paper under tension, and specular gloss at 75 deg.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee IV on Physical Test Methods for Container Board (M. J. Clark, chairman).—Close cooperation with the TAPPI Container Testing Group will result in early balloting on methods of test for burst, flat crush, and puncture.

Subcommittee VI, Editorial (W. R. Willets, chairman) is revising the Monograph on Paper and Paperboard: Characteristics, Nomenclature and Significance of Tests (*ASTM STP No. 60-A*) which is well along toward publication.

Subcommittee VII, ISO/TC6 on Paper (G. L. Adams, chairman), is made up of the Committee D-6 representatives to the USA Advisory Group to ISO/TC6 on Paper. The USA Advisory Group is actively participating in the ISO subcommittee covering nomenclature, terminology, and substance, on test methods and quality specifications and in determining dimensions. Representatives recently attended ISO meetings in Paris and Lisbon.

Current matters under consideration are size of correspondence envelopes and pockets and a method for determination of moisture content of paper and board (oven drying method).

This report has been submitted to letter ballot of the committee, which consists of 85 voting members; 60 members returned their ballots, of whom 52 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. A. BIRDSALL,
Chairman.

R. E. GREEN,
Secretary.

REPORT OF COMMITTEE D-7
ON
WOOD*

Committee D-7 on Wood and nine of its subcommittees met at the Forest Products Laboratory, Madison, Wis., on Jan. 19 and 20, 1961.

RESEARCH ACTIVITIES

The major undertaking that has dominated research in Committee D-7 for the past six years has been the ASTM Wood Pole Research Program. Sponsored by ASTM and implemented by contributions of some $300,000 from 70 companies, organizations, agencies, and societies, the program culminated with the publication of the final report in 1961. The study involved tests of 620 treated and untreated poles of 5 species and over 14,000 tests of matched small clear specimens of wood. The basic purpose was to promote the more efficient utilization of wood poles through more extensive and reliable knowledge of their strength properties and of factors that affect these properties. The major effort during the past year comprised the compilation and analysis of all the data and its integration into the final report. The report, entitled "Strength and Related Properties of Wood Poles," by Lyman W. Wood, E. C. O. Erickson, and A. W. Dohr (with Foreword by L. J. Markwardt) (*STP No. 295*) may be purchased from ASTM at a nominal price.

At the meeting of Committee D-7 on January 20, 1961, the following resolution was unanimously adopted:

> Inasmuch as the ASTM Wood Pole Research Program involving the scientific strength testing of hundreds of full-sized poles and thousands of small specimens has been satisfactorily concluded, the data analyzed, and a comprehensive report of the entire test program just released, BE IT RESOLVED that Committee D-7, in session at Madison this January 20, acknowledges with most sincere thanks the services rendered by the Forest Products Laboratory, its Directors—two served during the course of the tests,—and its entire staff. Particularly do we salute L. J. Markwardt, R. P. A. Johnson, Lyman W. Wood, E. C. O. Erickson, and A. W. Dohr. This was a stupendous achievement well done.

One extensive study that has been under way for several years is the specific gravity or density survey of the forest resources, involving the important commercial species. This is a 10-yr project sponsored by the Forest Service and industry. It involves extensive tree sampling by the increment core method on a statistical basis and has as its object the evaluation of tree quality and species characteristics as influenced by environmental conditions and genetics. Substantial data have already been obtained on the southern yellow pines and work is being extended to cover western species. The results are of particular interest to Subcommittee I on Specifications for Timber.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

Another study under way by industry involves the mill sampling and testing of Douglas fir from a large number of operations within the range of commercial production, particularly the inland regions. Results are expected during the coming year that will be of particular interest to Committee D-7 in its review and revision of present standards involving basic stresses for different species.

NEW TENTATIVES

The committee recommends for publication as tentative the following proposed specifications and method as appended hereto:[1]

Tentative Specifications for:

Petroleum for Blending with Creosote, and Creosote - Petroleum Solution.

Tentative Method of Test for:

Moisture and Creosote-Type Preservative in Wood.

REVISION OF TENTATIVE

The committee recommends revisions as follows of the Tentative Specifications for Pressure Treatment of Timber Products (**D 1760 - 60 T**)[2] and continuation of the specifications as tentative.

Section 4(d).—Change Paragraphs (*1*) and (*2*) to read as follows:

(*1*) For empty-cell treatment the material shall be subjected to atmospheric (Lowry process) or to higher air pressures (Rueping process) of the necessary intensity and duration. For full-cell treatment, the material shall be subjected to a vacuum of not less than 22 in. at sea level for not less than 30 min either before the cylinder is filled or during the period of heating in preservative. These initial conditions shall be applied prior to filling and shall be maintained while the cylinder is being filled with preservative.

(*2*) When refusal treatment is specified, material shall be treated by the full-cell process; the pressure and temperature during the pressure period shall be maintained constant or increased within a range consistent with good practice for the material being treated until the quantity of preservative absorbed is not more than the following percentages of the amount already injected: for Douglas fir and oak, 2 per cent in each of any two consecutive half hours; for all other species, ½ per cent in any half hour.

Section 5(a)(3).—Add at end of paragraph the ASTM designation of the solvent referred to.

In the last line, omit the word "heavy."

Section 6.—In the table, add the ASTM Designation D 1858 to identify the creosote-petroleum solutions.

Section 7.—Change the word "specimens" to read "pieces," in Paragraph (*e*).

Add a new Paragraph (*f*) to read as follows:

As an alternate method, preservative retention in a specified zone of the treated product may be determined by extraction or analysis. Retentions determined by extraction or analysis and specified in subsequent standards shall be minimum retention and no tolerances shall apply.

Table II.—Make additions as follows:

	Southern Pine Ponderosa Pine	
Results of treatment:		
Retention, min, lb per cu ft:	General Use	Coastal Waters
Creosote and creosote solutions:		
Creosote		
By gage or scale weight.....	12	20
By extraction...		18
Zone, in. from surface.....		0 to 3
Number of borings.....		20
ASTM analytical Method....		D 1858

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[1] The new tentatives appear in the 1961 Book of ASTM Standards, Part 6.

[2] 1960 Supplement to Book of ASTM Standards, Part 6.

Table III.—Make additions as follows:

	Southern Pine
Results of treatment:	
Retention, min, lb per cu ft:	
Creosote and creosote solution:	
Cresote:	
By gage or scale weight	8, 10, 12[c]
By extraction:	6, 7.5, 9[c]
Zone, in. from surface	½ to 2
Number of borings	20
ASTM Analytical Method	D 1858

The retentions for creosote by gage or scale weight presently shown shall apply for all other species.

Change penetration requirement for lodgepole pine poles from "1.5 or 85" to "½ and 85 up to a maximum of 1.5."

Table IV.—Change retention for Black or Red Gum ties from "8" to "10" lb per cu ft for all preservatives presently recommended.

Table V.—Change retention for Jack Pine posts for creosote-petroleum solution from "7" to "6" lb per cu ft.

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends revisions as indicated of the Standard Specification for Creosote-Coal Tar Solution (D 391 – 53)[3] and reversion of the specification to tentative.

Table I.—Revise as shown in the accompanying Table I.[4]

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for:

Acid Copper Chromate (**D 1624 – 59 T**),[5]
Chromated Copper Arsenate (**D 1625 – 59 T**),[5] and
Chromated Zinc Arsenate (**D 1626 – 59 T**).[5]

Tentative Methods for:

Chemical Analysis of Acid Copper Chromate (**D 1627 – 59 T**),[5]
Chemical Analysis of Chromated Copper Arsenate (**D 1628 – 59 T**),[5]
Chemical Analysis of Chromated Zinc Arsenate (**D 1629 – 59 T**),[5] and
Testing Wood Preservatives by Laboratory Soil-Block Cultures (**D 1413 – 56 T**).[3]

WITHDRAWAL OF STANDARDS

The committee recommends the withdrawal of the following standards because zinc chloride is no longer of significant commercial importance in the wood-preserving industry:

Standard Methods of Chemical Analysis of Zinc Chloride (**D 199 – 50**),[3] and
Standard Specifications for Zinc Chloride (**D 432 – 50**).[3]

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Standard Specifications for:

Creosote (**D 390 – 53**), and
Chromated Zinc Chloride (**D 1032 – 50**)

Standard Methods of:

Sampling and Testing Creosote (**D 38 – 33**),
Volume and Specific Gravity Correction Tables for Creosote and Coal Tar (**D 347 – 33**),
Test for Benzene-Insoluble Matter in Creosote (**D 367 – 49**),
Test for Specific Gravity of Creosote (**D 368 – 33**),
Test for Specific Gravity, 38/15.5 C, of Creosote Fractions (**D 369 – 33**),
Chemical Analysis of Chromated Zinc Chloride (**D 1033 – 50**),

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[3] 1958 Book of ASTM Standards, Part 6.

[4] Table I is not included in this report but appears in the revised specification; see 1961 Book of ASTM Standards, Part 6.

[5] 1959 Supplement to Book of ASTM Standards, Part 6.

Chemical Analysis of Tanalith (**D 1035 - 50**),
Testing Small Clear Specimens of Timber (**D 143 - 52**),
Static Tests of Timbers in Structural Sizes (**D 198 - 27**),
Test for Tar Acids in Creosote and Creosote-Coal Tar Solutions (**D 453 - 41**), and
Testing Veneer, Plywood, and Other Glued Veneer Constructions (**D 805 - 52**).

Standard Definitions of Terms Relating to:

Timber Preservatives (**D 324 - 41**),
Veneer and Plywood (**D 1038 - 52**),
Timber (D 9-30), and
Domestic Hardwoods and Softwoods (**D 1165 - 52**).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Specifications for:

Wooden Paving Blocks for Exposed Platforms, Pavements, Driveways, and Interior Floors Exposed to Wet and Dry Conditions (**D 52 - 59 T**).

Tentative Methods of:

Establishing Structural Grades of Lumber (**D 245 - 57 T**), and
Conducting Machining Tests of Wood and Wood-Base Materials (**D 1666 - 59 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[6]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Specifications for Timber (L. W. Wood, chairman).—The principal activity of the subcommittee during the year has been that of the Task Group on Basic Stresses. The group, consisting of R. R. Cahal, R. E. Eby, L. J. Markwardt, W. A. Oliver, and L. W. Wood, has held three meetings of two to three days each and has given much thought and discussion to the revision of basic stresses in Method D 245. The group has available a great body of data from the Forest Products Laboratories of the United States and Canada and is expecting additional information from the Western Pine Association and the U. S. Forest Service. Virtual agreement has been reached on the general reduction factors to be used to convert species average values to basic stresses. Natural marketing groups of species are recognized. Although a number of specific values of basic stresses have been considered and discussed, the Task Group is not yet ready to present these values to Subcommittee I. Because of the importance of this assignment and the urgency of completing it as rapidly as possible, while at the same time giving full consideration to all data bearing on the recommendations, the following statement was agreed upon:

> The Task Group has had at its disposal the extensive accumulated data from the Forest Products Laboratories of the United States and Canada, and from industry and other sources, and further welcomes the presentation of additional data as they become available for use in the revision of ASTM Method D 245.
>
> In reviewing its assignment, it recognizes that all ASTM standards are subject to continual review and revision, and that any draft recommendations to be made, whether forthcoming in 2 years or in 10 years, would need to be reviewed periodically under ASTM procedure.
>
> Considering the importance of completing the assignment on basic stresses, and with this concept in mind, the Task Group is anticipating the opportunity of reviewing forthcoming data from current mill sampling tests and from the Forest Service-industry specific gravity survey before completing its initial draft recommendations within about 1 or 2 years, as a target date.

A detailed proposal was received for recommended practice in establishing working stresses for load-sharing members such as joists and rafters. The proposal calls for determining working stresses by applying suitable reduction factors to strength values obtained by surveys in any grade of lumber to arrive at working stresses without the use of basic stresses or strength ratios. It was discussed in subcommittee at a meeting on Jan. 19 and was referred to a Task Group for study, with the subcommittee chairman authorized to submit the recommendation of the Task Group to the subcommittee at his discretion.

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[6] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee V on Methods of Preservative Treatment of Timber (C. W. Best, chairman).—Last year the subcommittee developed a new Tentative Specification for Pressure Treatment of Timber Products (D 1760 - T). Further review of the tentative during the year resulted in the recommendation of some editorial changes to clarify certain sections and some revisions. Details of these changes and the action recommended are presented earlier in this report.

Subcommittee VI on Timber Preservatives (D. L. Davies, chairman).—The subcommittee has developed and approved three new tentatives appended to this report: (1) Specification for Petroleum for Blending with Creosote, (2) Specification for Creosote-Petroleum Solution, and (3) Method of Test for Moisture and Creosote-Type Preservative in Wood. Included also in the recommendations is a revision of Specification D 391 with reversion to tentative status. The revision consists in a change in Table I. Because zinc chloride is no longer of commercial importance as a wood preservative, the subcommittee is recommending the withdrawal of the two standards (D 199 - 50 and D 432 - 50) relating to this product.

Subcommittee VII on Wood Poles and Cross Arms (R. P. A. Johnson, chairman).—The subcommittee reports the completion of the ASTM Wood Pole Research Program as noted earlier in this report. It is distributed to American Standards Association Committee 05 and will be useful to it in its consideration and revision of "American Standard Specifications and Dimensions of Wood Poles."

In a meeting on Jan. 19, the subcommittee recognized the existence of further problems within its scope of activity and intends to remain intact and to work on those problems. Among these are methods of test from cross arms and study of the testing and specification of wood poles for building framing. Poles and crossarms are important uses of wood, and the subcommittee believes that they are worthy of continuing attention by Committee D-7.

Subcommittee IX on Methods of Testing (Alan D. Freas, chairman).—The subcommittee discussed at considerable length a suggested procedure for obtaining samples for tests of small clear specimens.

The subcommittee discussed the desirability of developing a set of definitions pertaining to mechanical fastenings for wood. It was generally agreed that such definitions would be desirable, but there was lack of agreement as to scope and detail. A task committee will be appointed to consider these questions and to develop one section of such a compilation for consideration by the subcommittee.

In addition to the development of definitions for mechanical fastenings, it is planned that, during the next year, work will be undertaken on standards for methods of determining specific gravity of wood and for methods of simulated service testing for flooring of wood or wood-base materials.

Subcommittee XI on Moisture Content of Timber (R. C. Rietz, chairman).—The subcommittee has reviewed and revised portions of the proposed wood moisture determination methods prepared by a Task Group. Two methods: the oven testing method, and electrical methods, were reviewed, and considerable progress made on the revisions of these writeups for inclusion in the proposed standard. The distillation and hygrometric methods portions of the standard will soon be available for inclusion and a critical review by the members of the subcommittee is expected to be completed prior to next year's meeting of Committee D-7.

Subcommittee XII on Fire-Retardant Wood (H. O. Fleischer, chairman).—The subcommittee met at the Forest Products Laboratory on the afternoon of January 19, 1961, with approximately 25 in attendance. A film was presented on recent wood and steel roof framing tests, conducted at Southwest Research Laboratories in San Antonio, Tex. under the sponsorship of the National Lumber Manufacturers Association. Another film was presented on the St. Lawrence Seaway burn-out tests conducted by the Division of Building Research, National Research Council of Canada.

The subcommittee discussed the industry's need for a fire test method for assemblies and constructions that would combine measurement of flame spread, fuel contributed, and structural performance. The importance of joints and fastenings was brought out in the discussion, and it was felt that a test as proposed might be particularly useful in this respect. A Task Group was appointed to explore the need for such a new test, to assemble further information on the subject, and to report to the subcommittee at its next meeting.

The subcommittee also discussed the difficulties encountered in the use of Method E 119, which limits the use of wood in applications where a 250 F temperature rise occurs on the unexposed side of a wall or barrier. It was agreed that there is need for a review of the applicable data on the fire performance of wood in structures, and that new data will no doubt need to be developed, before the question should be brought to the attention of Committee E-5 on Fire Tests of Materials and Constructions.

Subcommittee XIII on Durability and Exposure (R. M. Lindgren, chairman).—A draft of an accelerated laboratory method of evaluating natural decay in wood was presented to the subcommittee. The Task Force having this assignment will refine this method during the coming year and present a revised draft for review at its next meeting.

Plans were made for the preparation of two additional methods: field evaluation of preservatives in posts; and accelerated methods of evaluating wood preservatives against marine borers. Work on these will continue during the year.

Subcommittee XV on Wood Base Fiber and Particle Panel Materials (Wayne C. Lewis, chairman).—Active interest continues in the work of Subcommittee XV and several important problems are under consideration by a number of Task Groups. One Task Group has under consideration the needs for specifications for particle board. Another active Task Force is that on structural insulating roof deck. The work of this group has required liaison between Subcommittee XV and Committee C-16 on Thermal Insulating Materials. It has been understood in this connection that the development of any specific procedures relating to strength properties should be within the province of Committee D-7 and that Subcommittee II of C-16 will otherwise continue its activity developments. Other Task Groups cover subject matter as follows: (1) the development for a specification for hardboard, (2) the development of test procedures for underlayment, (3) the development of specifications for high-density board for specific building applications, and (4) revisions of Methods D 1037. In Method D 1037, particular consideration is given to the revision of the procedure for measuring linear expansion to a compression-parallel-to-surface test procedure and to a compression-perpendicular-to-surface test. Another Task Group has under study the possible need of revisions of Definitions D 1554.

This report has been submitted to letter ballot of the committee, which consists of 104 members; 86 members returned their ballots, of whom 81 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. J. MARKWARDT,
Chairman.

W. A. OLIVER,
Secretary.

REPORT OF COMMITTEE D-8
ON
BITUMINOUS MATERIALS FOR ROOFING, WATERPROOFING, AND RELATED BUILDING OR INDUSTRIAL USES*

Committee D-8 on Bituminous Materials for Roofing, Waterproofing, and Related Building or Industrial Uses held two meetings during the year: in Atlantic City, N. J., on June 29, 1960, and in Cincinnati, Ohio, on Feb. 3, 1961.

The membership consists of 88 voting members, of whom 48 are classified as producers, 22 as consumers, and 18 as general interest members.

The committee suffered loss through the death of Carl Bussow, and T. I. Coe, and through the resignation of J. G. Hayes, J. E. Boarini, H. A. Pasman, J. Weisberg, A. B. Brown, R. B. Dull, D. F. Fink, A. J. Hoiberg, E. M. Johnson, and A. D. MacNutt.

NEW TENTATIVES

The committee recommends for publication as tentative the following specifications and methods as appended hereto:[1]

Tentative Specifications for:

Homogeneous Bituminized Fiber Drain and Sewer Pipe,
Laminated-Wall Bituminized Fibre Drain and Sewer Pipe, and
Mineral Aggregate for Use on Built-Up Roofs.

Tentative Method of Test for:

Moisture in Mineral Aggregate for Use on Built-Up Roofs,
Hardness of Mineral Aggregate for Use on Built-Up Roofs, and
Translucency of Mineral Aggregate for Use on Built-Up Roofs.

REVISIONS OF TENTATIVE

The committee recommends revisions as indicated of the following tentative and its continuation as tentative:

Tentative Specifications for Woven Glass Fabrics Saturated with Bituminous Substances for Use in Waterproofing (D 1668 – 59 T):[2]

Title.—Substitute the word "treated" for the word "saturated."

Section 2(a) and (b).—Change the beginning of the first sentence of both Paragraphs (*a*) and (*b*) to read: "For the plying cement employed in the construction . . ."

TENTATIVE REVISIONS OF STANDARDS

The committee recommends tentative revision as indicated of the following standards:

Standard Specifications for Primer for Use with Asphalt in Dampproofing and Waterproofing (D 41 – 41):[3]

Section 2(a).—Change the Furol viscosity requirement from "25 to 150 sec" to read "25 to 75 sec."

Section 4.—Add a new Paragraph (*e*) to read as follows: "(*e*) *Water.*—Method of Test for Water in Petroleum Products and Other Bituminous Materials (ASTM Designation: D 95)."

*** Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.**

[1] The new tentatives appear in the 1961 Book of ASTM Standards, Part 4.

[2] **1959 Supplement to Book of ASTM Standards, Part 4.**

[3] **1958 Book of ASTM Standards, Part 4.**

Standard Specifications for Asphalt Shingles Surfaced with Mineral Granules (D 225 – 60):[4]

Table I.—Add a new footnote to read: "Minimum weight of weatherside coating shall be increased to 23.5 lb when the shingles are embossed to produce a textured or grained effect."

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the revision as appended hereto[5] of the Standard Method of Test for Steam Distillation of Bituminous Protective Coatings (**D 255 – 28**), and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

WITHDRAWAL OF STANDARD

The committee recommends the withdrawal of the Standard Specifications for Sieve Analysis of Granular Mineral Surfacing for Asphalt Roofing and Shingles (**D 1001 – 51**).

STANDARDS CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following standards under the joint jurisdiction of Committees D-4 and D-8:

Standard Methods of Sampling Bituminous Materials (**D 140 – 55**), and
Standard Volume Correction Table for Tar and Coal Tar Pitch (**D 633 – 44**).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Methods of:

Testing Asphalt Roll Roofing, Cap Sheets, and Shingles (**D 228 – 57 T**),
Testing Asphalt-Base Emulsions for Use as Protective Coatings for Built-Up Roofs (**D 1167 – 58 T**), and
Test for Failure End Point in Accelerated and Outdoor Weathering of Bituminous Materials (**D 1670 – 59 T**).

Tentative Recommended Practice for:

Accelerated Weathering Test of Bituminous Materials (**D 529 – 59 T**).

TENTATIVE REVISIONS CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the tentative revisions as indicated of the following standards:

Standard Methods of Testing Emulsified Asphalts (**D 244 – 60**): tentative revision issued June, 1958.
Standard Specifications for Asphalt-Saturated and Coated Asbestos Felts for Use in Constructing Built-Up Roofs (**D 655 – 47**): tentative revision issued June, 1957, and 1959.

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following standards and tentative:

Standard Specifications for Primer for Use with Asphalt in Dampproofing and Waterproofing (D 41 – 41):[3]

Section 3(b).—In line 10, insert the word "to" after the word "prior."

Standard Method of Test for Staining Properties of Asphalts (Modified Pressure Method) (D 1328 – 58):[3]

Section 2(f).—Change to read as follows:

> The grade of cigarette paper to be used shall be agreed upon by the asphalt consumer and the asphalt supplier. The paper used by different laboratories for comparative tests shall be obtained from the same source and shall be from the same lot.[3]

Footnote 3.—Change to read: "Emil

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[4] 1960 Supplement to Book of ASTM Standards, Part 4.

[5] The revised standard appears in the 1961 Book of ASTM Standards, Part 4.

Greiner, 2026 N. Moore St., New York 13, N. Y., can supply quantities of Washington No. 4 cigarette paper for this test."

Tentative Method for Preparation of Test Panels for Accelerated and Outdoor Weathering of Bituminous Coatings (D 1669 – 59 T):[2]

Section 2(a)(1).—Change the aluminum alloy designation now reading "3S-H14" to read "No. 3003–H14."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[6]

This report has been submitted to letter ballot of the committee, which consists of 95 members; 85 returned their ballots, of whom 72 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. R. SNOKE,
Chairman.

H. B. KEENE,
Secretary.

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[6] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-8 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Method of Test for:

Sieve Analysis of Nongranular Mineral Surfacing for Asphalt Roofing and Shingles (D 452 – 60 T).

Revision and Reversion to Tentative of Standard Method of Test for:

Sieve Analysis of Granular Mineral Surfacing for Asphalt Roofing and Shingles (D 451 – 40).

These recommendations were accepted by the Standards Committee on September 12, 1961, and the revised tentative methods appear in the 1961 Book of ASTM Standards, Part 4.

REPORT OF COMMITTEE D-9
ON
ELECTRICAL INSULATING MATERIALS*

Committee D-9 on Electrical Insulating Materials held three meetings during the year: in Atlantic City, N. J., June 27–29, 1960, during the Annual Meeting of the Society; in Detroit, Mich., Oct. 5–7, 1960; and in Louisville, Ky., March 6–8, 1961.

At the time of the Louisville meeting, the total membership comprised 328, of whom 213 were classified as official voting representatives, made up of 93 producers, 73 consumers, and 47 general interest members.

No major organizational changes have been made during the year. The proposed formation of a new Subcommittee XVI on Thermal Endurance, previously announced, has been abandoned. The proposal to form a new subcommittee on dielectric films was also cancelled, but this work is to be covered by a new Section G of Subcommittee VII.

Committee D-9 has continued its cooperation with other groups concerned with electrical insulation, including the International Electrotechnical Commission (IEC), the International Organization for Standardization (ISO), the American Institute of Electrical Engineers (AIEE), the Electronic Industries Assn. (EIA), and the National Electrical Manufacturers Assn. (NEMA). The committee has continued its participation in the technical programs of the AIEE-NEMA Electrical Insulation Conference (formerly National Conference on the Application of Electrical Insulation). In November, 1960, A. H. Scott, K. N. Mathes, and J. F. Dexter attended IEC/TC 15 meetings in New Delhi, India.

The committee held a symposium at the meeting in October, 1960, on the subject of "Insulation Resistance—Its Meaning and Significance," under the sponsorship of Subcommittees XI and XII.

Recommendations Accepted by the Administrative Committee on Standards

Subsequent to the 1960 Annual Meeting, Committee D-9 presented to the Society through the Administrative Committee on Standards the following recommendations which were accepted on the dates indicated:

New Tentative Methods of Test for:

Electrical Resistance of Ceramic Materials at Elevated Temperatures (D 1829 – 61 T) (Accepted May 10, 1961).

Thermal Stability of Electrical Insulating Coated Fabrics by Curved Electrodes (Dielectric Breakdown Method) (D 1830 – 61 T) (Accepted May 10, 1961).

New Tentative Recommended Practice for:

Etching and Cleaning Thermosetting Copper-Clad Laminates for Electrical Testing (D 1825 – 61 T) (Accepted May 10, 1961).

Revisions of Tentative Methods of Sampling and Testing:

Untreated Paper Used for Electrical Insulation (D 202 – 55 T) (Accepted Sept. 29, 1960, and May 10, 1961).

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* **Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.**

Revision of Tentative Methods of Testing:

Vitrified Ceramic Materials for Electrical Applications (D 116 – 59 T) (Accepted Sept. 29, 1960),

Nonrigid Vinyl Chloride Polymer Tubing (D 876 – 60 T) (Accepted May 10, 1961), and

Pressure-Sensitive Adhesive Coated Tapes Used for Electrical Insulation (D 1000 – 60 T) (Accepted May 10, 1961).

Revision of Tentative Specifications for:

Electrical Insulating Paper—Interlayer Type (D 1305 – 54 T) (Accepted Sept. 29, 1960), and

Natural Muscovite Mica Based on Visual Quality (D 351 – 57 T) (Accepted Sept. 29, 1960).

Revision of Tentative Recommended Practice for:

Exposure of Polymeric Materials to High Energy Radiation (D 1672 – 59 T) (Jointly with Committee D-20) (Accepted May 10, 1961).

The revisions of Tentative Methods D 116 and D 202 and the revisions of Tentative Specifications D 351 and D 1305 which were accepted by the Administrative Committee on September 29, 1960, appear in the 1960 Supplement to Book of ASTM Standards, Part 9. The other items are available as separate reprints.

NEW TENTATIVES

The committee recommends for publication as tentative the following method and specification as appended hereto or otherwise indicated:[1]

Tentative Method for:

Corona Measurement, as published in Appendix IV in the 1957 edition of the compilation of "ASTM Standards on Electrical Insulating Materials," and issued later as a separate reprint.

Tentative Specifications for:

Copper-Clad Thermosetting Laminates for Printed Wiring.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Specifications for Vulcanized Fibre Sheets, Rods, and Tubes Used for Electrical Insulation (D 710 – 54 T):[2] Revise as appended hereto.[3]

Tentative Methods of Testing Vitrified Ceramic Materials for Electrical Applications (D 116 – 60 T):[4]

Section 4.—Revise the second sentence to read: "It indicates quality and consistency of quality."

New Section 8.—Add a new Section 8 under Dielectric Strength entitled "Significance," to read as follows, renumbering the present Section 8 as Section 9:

> 8. Dielectric strength of a ceramic is of importance in comparing different materials or controlling quality of different lots. The values obtained usually will have little relation to the dielectric strength realized in service. While mechanical requirements often dictate thickness of dielectrics far greater than needed to withstand the electrical stress, dielectric strength data will serve as a guide in estimating the electrical safety factor.

New Section 10.—Add a new Section 10 under Thermal Shock entitled "Significance," to read as follows, renumbering the present Section 9 as Section 11, and renumbering subsequent sections accordingly:

> 10. Thermal shock tests may serve as an evaluation of the resistance of a particular ceramic composition, shape, and dimension to temperature stress relative to another composition of the same shape and dimension. They also permit the determination of the resistance of a given ceramic material to simulated environmental heat service conditions, and they make possible the evaluation of shape or design

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[1] The new tentatives appear in the 1961 Book of ASTM Standards, Part 11.

[2] 1958 Book of ASTM Standards, Part 9.

[3] The revised tentative appears in the 1961 Book of ASTM Standards, Part 11.

[4] 1959 Supplement to Book of ASTM Standards, Part 9.

factors as they might affect normal thermal resistance. It is presupposed that test conditions are sufficiently extreme to cause structural failures.

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends revisions as appended hereto[5] of the Standard Method of Testing Sheet and Plate Materials Used for Electrical Insulation (D 229 – 58)[2] and reversion of the Method to tentative.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision in substance:

Tentative Specifications for:

Orange Shellac and Other Indian Lacs for Electrical Insulation (**D 784–59 T**), and

Absorbent Laminating Paper for Electrical Insulation (**D 1080 – 58 T**).

Tentative Methods of:

Test for High-Voltage, Low-Current Arc Resistance of Solid Electrical Insulating Materials (**D 495 – 58 T**), with editorial changes in Sections 2, 3, and 6,

Testing Nonrigid Vinyl Chloride Polymer Tubing (**D 876 – 60 T**), with editorial changes in Sections 19 and 20,

Testing Pressure-Sensitive Adhesive Coated Tapes Used for Electrical Insulation (**D 1000 – 61 T**),

Testing Hydrocarbon Waxes Used for Electrical Insulation (**D 1168 – 58 T**),

Test for Dielectric Constant and Dissipation Factor of Polyethylene by Liquid Displacement Procedure (**D 1531 – 59 T**),

Test for Dielectric Constant and Dissipation Factor of Expanded Cellular Plastics Used for Electrical Insulation (**D 1673 – 59 T**), and

Testing Polymerizable Embedding Compounds Used for Electrical Insulation (**D 1674 – 59 T**).

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee recommends that the tentative revisions as indicated of the following standards be approved for reference to letter ballot of the Society for adoption as standard:

Standard Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (**D 618 – 58**)[2] (Jointly with Committee D-20 on Plastics): Sections 3, 4, 8, and new Section 4, issued 1959.

Standard Methods of Test for Electrical Resistance of Insulating Materials (**D 257 – 58**):[2] Sections 3, 4, and Appendix I, issued November, 1959.

Standard Methods of Test for Dielectric Breakdown Voltage and Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (**D 149 – 59**):[4] Section 2 and Appendix I, issued November, 1959.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Methods of Testing Varnished Cotton Fabrics and Varnished Cotton Fabric Tapes Used for Electrical Insulation (D 295 – 58):[2]

New Section on Tearing Strength.—Add the following new Sections 19 to 24 under the centered heading "Tearing Strength," renumbering the present Sections 19 to 24 and subsequent sections accordingly.[6]

Present Sections 37 to 43.—Change the centered heading to read "Dielectric Breakdown Voltage Under Elongation," and retain the present Note. Add Sections 37 and 38 as follows.[6]

Retain present Sections 39, 40, and 41. Delete the Note at the end of Section 42.

Change Section 43 to read as follows:

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[5] The revised method appears in the 1961 Book of ASTM Standards, Part 11.

[6] The new sections are not included in this report but appear in the revised methods; see 1961 Book of ASTM Standards, Part 11.

“Report

“43. The report shall include the following:

“(*1*) The average dielectric breakdown voltage in kilovolts on the elongated material,

“(*2*) The percentage elongation during the test,

“(*3*) The relative humidity and temperature during the conditioning period and at the time of test, and

“(*4*) The specimen thickness in mils before and under elongation.”

TABLE I.—DIELECTRIC BREAKDOWN REQUIREMENTS.[a]
(Revision of Table I, Specifications D 922)

Wall Thickness, in.	Dielectric Breakdown for Grades A and C, kv	Dielectric Breakdown for Grade B, kv
0.012	9.0	7.7
0.016	12.5	9.4
0.020	14.0	10.6
0.025	15.7	11.7
0.030	17.1	12.6
0.035	18.0	13.5
0.040	19.2	14.4
0.045	20.4	15.3
0.050	21.5	16.2
0.055	22.8	17.1
0.060	24.0	18.0

[a] For a wall thickness not listed in this table, the dielectric breakdown value shall be that prescribed for the nearest smaller thickness.

Standard Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 – 58)[2] (Jointly with Committee D-20):

Section 3(d).—Add the following test temperatures and tolerances to the list in this paragraph:

Test Temperatures	Tolerance, plus or minus
225 C (437 F)	3.0 C (5.4 F)
275 C (527 F)	3.0 C (5.4 F)
325 C (617 F)	4.0 C (7.2 F)
350 C (662 F)	5.0 C (9.0 F)
400 C (752 F)	6.0 C (10.8 F)
450 C (842 F)	8.0 C (14.4 F)
500 C (932 F)	10.0 C (18.0 F)
600 C (1112 F)	12.0 C (21.6 F)

Standard Methods of Testing Varnished Glass Fabrics, and Varnished Glass Fabric Tapes Used for Electrical Insulation (D 902 – 56):[2]

Title.—Change to read, “Standard Methods of Testing Flexible Resin Coated Glass Fabrics and Glass Fabric Tapes Used for Electrical Insulation.”

Revise the text editorially changing “varnished glass fabrics” to read “resin coated glass fabrics” and changing “oleoresinous varnish glass fabric” to read “oleoresinous varnished coated glass fabrics.”

Section 40(a).—Change to read as follows:

(*a*) With half the test specimens in position, the specimen-holding fixtures shall be placed in the oven which has been previously brought to the required baking temperature. For silicone resin coated fabrics intended for use in AIEE Class H applications the temperature shall be 250 ± 3 C (482 ± 5.4 F), or as otherwise agreed upon by the purchaser and the supplier; for resin coated fabrics intended for use in AIEE Class F applications the temperature shall be 180 ± 3 C (356 ± 5.4 F).

NOTE.—Thermal stability of these fabrics may be determined by the Methods of Test for Thermal Stability of Electrical Insulating Coated Fabrics by Curved Electrodes (Dielectric Breakdown Method) (ASTM Designation: D 1830).

Standard Specifications for Nonrigid Vinyl Plastic Tubing (D 922 – 59):[2]

Section 4.—In the first sentence, delete the phrase “except sizes Nos. 24 and 22.”

Revise Paragraphs (*h*) and (*i*) to read as follows and revise Table I as shown in the accompanying Table I:

(*h*) *Dielectric Breakdown*.—The average dielectric breakdown shall not be less than that prescribed for the corresponding wall thickness in Table I.

(*i*) *Dielectric Breakdown at High Humidity*.—The average dielectric breakdown strength at 96 per cent relative humidity shall not be less than 90 per cent for Grade A and 85 per cent for

Grades B and C of the dry value obtained on test.

Standards Continued Without Revision

The committee recommends the continuation without revision of the following standards:

Standard Methods of:

Testing Varnishes Used for Electrical Insulation (**D 115 – 55**),
Testing Solid Filling and Treating Compounds Used for Electrical Insulation (**D 176 – 59**),
Test for Conducting Paths in Electrical Slate (**D 273 – 40**),
Testing Shellac Used for Electrical Insulation (**D 411 – 52**),
Measuring Shrinkage from Mold Dimensions of Molded Materials Used for Electrical Insulation (**D 551 – 41**),
Test for Product Uniformity of Phenolic Laminated Sheets (**D 634 – 44**),
Test for Tensile Strength of Molded Electrical Insulating Materials (**D 651 – 48**),
Measuring Dimensions of Rigid Tubes Used for Electrical Insulation (**D 668 – 52**), and
Measuring Dimensions of Rigid Rods Used for Electrical Insulation (**D 741 – 52**),
Test for Power Factor and Dielectric Constant of Natural Mica (**D 1082 – 54**).

Tentatives Continued Without Revision

The committee has reviewed all other tentatives under its jurisdiction that have been published for two years or longer without revision and recommends their continuation without revision.

Editorial Change

The committee recommends editorial change as follows in the Tentative Methods of Testing Hydrocarbon Waxes Used for Electrical Insulation (**D 1168 – 58 T**):[2]

Section 2.—In Table I and Paragraph (*a*), change the reference to color method from Method D 155 which has been discontinued to Method D 1500.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[7]

Activities of Subcommittees

Subcommittee I on Insulating Varnishes, Paints, and Lacquers (W. E. Harvey, chairman) has developed a proposed method of test for the evaluation of thermal stability of flexible electrical insulating varnishes, and is working on a modification of the test for set time of phenol-formaldehyde varnishes. The subcommittee is also determining interest in work on "fluidized bed" coatings.

Subcommittee II on Effects of Nuclear and High Energy Radiation (D. J. Metz, chairman).—This joint D-9/D-20 subcommittee was responsible for the revision of Recommended Practice D 1672 noted earlier in this report as one of the recommendations accepted by the Administrative Committee on Standards. The subcommittee has continued its efforts to promote close coordination of its activities with those of other groups.

Subcommittee III on Plates, Tubes, Rods, and Molded Materials (Kenneth Wechsler, chairman) has completed a revision of the Specifications D 709 which has been approved by D-9 letter ballot but awaits Committee D-20 action for final approval. The subcommittee has developed the specifications for copper-clad laminates and the recommended practice for etching and cleaning these materials for electrical testing; and has revised Specifications D 710 and Method D 229.

Subcommittee V on Ceramic Products (W. W. Perkins, chairman) developed the Tentative Method of Test for Electrical Resistance of Ceramics at High Temperatures, which has been accepted by the Administrative Committee on Standards. The subcommittee has under consideration further additions to the

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[7] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Methods D 116. The methods of test and specifications for pin-type lime-glass insulators are being studied. Investigations into dielectric strength and dielectric constant and dissipation factor in high and low frequency ranges are under way. A study of electrodes for d-c resistance and a-c dielectric property testing is to be conducted.

Subcommittee VI on Solid Filling, Treating, Encapsulating, and Embedding Compounds (Thomas Hazen, chairman).—The section on polymerizable embedding compounds has conducted a limited interlaboratory test on the use of embedded electrodes for dielectric strength testing. Work has also continued on the preparation of test methods for dielectric constant and dissipation factor of polymerizable embedding materials.

Subcommittee VII on Flexible Sheet, Tape, and Tubing (F. P. West, chairman) is working on test methods for self-fusing tapes, silicone rubber tubing, heat shrinkable tubing, zipper tubing, and polymeric film; and on a specification for polytetrafluoroethylene tubing. In connection with thermal stability testing of Class F coated fabrics, recommended changes in Specifications for Gravity-Convection and Forced-Ventilation Laboratory Ovens (E 145) with respect to Type II, Grade B, ovens have been agreed upon and a number of ovens will be checked against the revised specifications. A revision of Methods D 902 and D 350, Specifications D 372, and a new specification for fully-cured silicone rubber coated glass cloth and tape have been prepared for Committee D-9 ballot. Specifications for black and yellow straight-cut and bias-cut varnished cloth and tape have been revised and submitted to subcommittee letter ballot.

Subcommittee VIII on Insulating Papers (P. L. Staats, chairman) was responsible for the revision of Methods D 202 which has been accepted by the Administrative Committee on Standards. A further revision of Method D 202, relating to the methods of determination of acidity-alkalinity-pH of untreated electrical insulating papers has been prepared and submitted to Committee D-9 letter ballot. The subcommittee has also developed a proposed specification for kraft dielectric tissue, capacitor grade, and has submitted it to committee letter ballot. Activities are continuing on methods for chloride determination, conductivity, holes, felt-hair cuts, and tests for impregnated paper specimens.

Subcommittee IX on Mica Products (K. G. Coutlee, chairman) was responsible for the revision of Specifications D 351 which was accepted by the Standards Committee. An important contribution by the subcommittee has been made by undertaking the preparation of mica standards for the Indian Standards Institution. Some work is being considered leading to the eventual preparation of ASTM specifications for mica splittings.

Subcommittee X on Magnet Wire Insulation (K. N. Mathes, chairman) has made considerable progress on test methods for film-coated wire and on a thermal aging specification. The subcommittee held a special meeting in New York City on Feb. 17, 1961, to expedite its work.

Subcommittee XI on Nomenclature, Significance, and Statistics (C. L. Craig, chairman) has continued to function as a review board for all new and revised methods and specifications to ensure conformity with correct ASTM usage. Considerable effort is being expended in connection with the standardization of definitions of electrical terms. A proposed tentative recommended practice for interlaboratory testing of electrical insulating materials has been prepared and submitted to D-9 letter ballot. The subcommittee is also working on a new recommended practice for sampling of electrical insulating materials. In cooperation with Subcommittee XII, the subcommittee assisted in the symposium on insulation resistance at the Detroit meeting in October, 1960.

Subcommittee XII on Electrical Tests (N. W. Edgerton, chairman) is completely revising the Methods of Test for A-C Capacitance, Dielectric Constant, and Loss Characteristics of Electrical Insulating Materials (D 150). Methods for corona resistance, medium power arc resistance, and electrostatic phenomena are in preparation. The subcommittee is planning an ASTM session on insulation resistance at the next Electrical Insulation Conference.

Subcommittee XIII on Composite Insulating Materials (L. J. Timm, chairman).—This relatively new subcommittee has reported that work is in progress preparatory to the writing of test methods for slot-cell insulation.

Subcommittee XIV on Conditioning (G. M. Armstrong, chairman) recommended the adoption of the 1959 tentative revisions of Methods D 618, and also that higher test temperatures be added to the list of recommended standard test temperatures in Methods D 618. At the Louisville meeting, Subcommittee XIV recommended that Committee D-9 propose to Sectional Committee C59 of the American Standards Assn. the recommendation that the Specification for Enclosures and Servicing Units for Tests Above and Below Room Temperature (D 1197) be adopted as American Standard. The work of Subcommittee XIV is a joint activity with Committee D-20 on Plastics.

Subcommittee XV on Research and Technical Papers (H. S. Endicott, chairman) has continued to keep the committee informed about the joint NEMA-EIA-ASTM research project on insulation resistance testing of printed circuit boards at the University of Delaware through the distribution of progress reports. The chairman has reported that Committee D-9 has been asked and has agreed to conduct a technical session on insulation resistance at the 1962 Electrical Insulation Conference. Papers on instrumentation, interpretation and significance, and systems have been proposed.

This report has been submitted to letter ballot of the committee, which consists of 213 members; 192 members returned their ballots, of whom 142 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

ARNOLD H. SCOTT,
Chairman.

THOMAS HAZEN,
Recording Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-9 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specifications for:

Kraft Dielectric Tissue, Capacitor Grade (D 1930 - 61 T), and

Fully Cured Silicone Rubber Coated Glass Fabric and Tapes for Electrical Insulation (D 1931 - 61 T).

New Tentative Methods of Test for:

Thermal Endurance of Flexible Electrical Insulating Varnishes (D 1932 - 61 T).

Revision of Tentative Specifications for:

Flexible Treated Sleeving Used for Electrical Insulation (D 372 – 53 T).

Revision of Tentative Methods of:

Testing Vitrified Ceramic Materials for Electrical Applications (D 116 – 60 T),
Sampling and Testing Untreated Paper Used for Electrical Insulation (D 202 – 60 T), and
Testing Flexible Treated Sleeving Used for Electrical Insulation (D 350 – 57 T).

These recommendations were accepted by the Standards Committee on September 12, 1961, and the new and revised tentative specifications and methods appear in the 1961 Book of ASTM Standards, Part 11.

REPORT OF COMMITTEE D-10

ON

PACKAGING*

Committee D-10 on Packaging held one meeting during the year: at Fort Belvoir, Va., on Oct. 5, 1960. The subcommittees also met in Chicago, Ill., on April 10, 11, and 12, 1960.

The committee consists of 99 voting members, of whom 34 are classified as producers, 37 as consumers, and 28 as general interest members.

At the meeting of the ASTM Board of Directors held on January 11, 1961, the title of the committee was changed from "Shipping Containers" to "Packaging." An additional statement to the scope was requested and accepted by the Board of Directors. The complete scope now reads as follows:

Scope.—Nomenclature, definitions of terms, test methods, performance specifications, and study of the effect of various factors influencing strength and serviceability relating to packaging, including packages, shipping containers, and pallets.

The responsibility for the development of standards for the basic materials used in packaging shall in general rest with the materials committees.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of all tentatives under its jurisdiction with the exception of Method D 775 – 57 T which is being recommended for adoption as standard.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Method of Drop Test for Shipping Containers (**D 775 – 57 T**)[1] be approved for reference to letter ballot of the Society for adoption as standard without revision.

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards that have stood without revision for six or more years:

Standard Methods of:

Compression Test for Shipping Containers (**D 642 – 47**),
Incline Impact Test for Shipping Containers (**D 880 – 50**),
Test for Water Vapor Permeability of Packages (**D 895 – 51**),
Test for Water Resistance of Containers by Spray Method (**D 951 – 51**),
Drop Test for Bags (**D 959 – 50**),
Drop Test for Cylindrical Shipping Containers (**D 997 – 50**),
Test for Penetration of Liquids into Submerged Containers (**D 998 – 51**), and
Testing Large Shipping Cases and Crates (**D 1083 – 53**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] 1958 Book of ASTM Standards, Part 6.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Definition of Terms (C. S. Macnair, chairman) will continue the addition of new terms to Definitions D 996, as well as the revision and updating of existing definitions.

Subcommittee II on Methods Appraisal (C. R. Gustafson, chairman) is considering a number of new areas of activity for Committee D-10. These include the possibility of developing test methods in the fields of inflatable dunnage, carload testing, non-skid applications, thermal properties of containers, aerosol cans, and shelf packages. In addition, present methods not under the jurisdiction of existing active subcommittees will be reviewed by Subcommittee II for possible study and revision.

Subcommittee III on Moisture and Water Vapor Resistance (H. A. Bergstrom, chairman) is conducting interlaboratory studies on the Method of Test for Water Vapor Permeability of Packages (D 895) to determine the accuracy and reproducibility of the method with the intention of revising it where necessary to effect improvement.

Subcommittee V on Revolving Drum Test (M. J. Clark, chairman) is conducting an interlaboratory test to determine the ability of revised Method D 782 to rank boxes of different grades in the same relative order. It is expected that this work will complete the study of this method.

Subcommittee VI on Interior Packing (C. E. Swanson, chairman) is rapidly progressing in the development of a creep test under the direction of T. J. Grabowski, and is investigating the possibility of developing a procedure for a dynamic vibration test. Work is actively continuing on the study of compatibility of cushioning materials. The Instrumentation Task Group, under the direction of A. H. McKinlay, continues to serve Subcommittee VI in proposals requiring instrumental measurements as part of test method development.

Subcommittee VII on Stacking Test (K. R. Marvin, chairman) is continuing the development of a method of test for compression under static load.

Subcommittee VIII on Vibration Test (A. M. Bak, chairman) is continuing the refinement of Method D 999 with emphasis on methods of reporting frequency, calibration, and response to external force of items within the package.

Subcommittee IX on Incline Impact Test (R. N. Maxson, chairman) has tabulated various factors requiring special study in the operation of the incline impact tester, and has appointed task groups to cover several of these areas. Steps will be taken to revise Method D 880 and revert it to tentative status with an explanatory note defining the limitations of the existing test method.

Subcommittee X on Exterior Packaging (H. W. Maas, chairman) is developing test methods for packaging tapes under the direction of C. E. Twitchell. Other areas of exterior packaging requiring development are under study, but have not yet been assigned to working groups.

This report has been submitted to letter ballot of the committee, which consists of 99 members; 94 members returned their ballots, of whom 90 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

K. W. KRUGER,
Chairman.

R. F. UNCLES,
Secretary.

REPORT OF COMMITTEE D-11

ON

RUBBER AND RUBBER-LIKE MATERIALS*

Committee D-11 on Rubber and Rubber-Like Materials and its subcommittees met twice during the year: in Atlantic City, N. J., on June 29 to July 1, 1960, and in Cincinnati, Ohio, on February 1 to 3, 1961.

Five technical papers were presented at the Atlantic City meeting as follows: "Good and Bad Effects of Nuclear Radiation on Rubber,"[1] by John W. Born of the B. F. Goodrich Research Center; "The Factor of Ozone Generation in Tests of Radiation Damage to Elastomers," by L. B. Bang, Goodyear Tire and Rubber Co., Research Division; "Dynamic Testing of Rubber Products Using a Sinusoidal Strain Machine," by A. R. Payne of the British Rubber Manufacturers Research Assn.; "The Tearing of Rubber," by A. G. Veith of the B. F. Goodrich Research Center; and "High-Temperature Immersion Testing of Elastomers," by C. T. Lenk, Wyandotte Chemical Co.

At the February meeting, John Mandel of the National Bureau of Standards presented a paper on "Interlaboratory Studies of Test Methods."

Considerable progress was made during the year on definitions for the terms "rubber," "rubber products," and "elastomer." A joint task group including members from both Committees D-11 and D-20, with R. E. Hess as chairman, met in Atlantic City on June 29, 1960, to review the definitions proposed by Committee D-11 which were then referred back to Subcommittee VIII on Nomenclature and Definitions with suggested revisions. The revised definitions were referred to the Society and the Administrative Committee on Standards accepted the definitions for the terms "rubber" and "rubber products" for publication in the Tentative Definitions D 1566 - 60 T. The definitions for the term, "rubber-like" was referred back to Committee D-11 for further consideration. The joint task group met again in New York City on December 8, 1960, and recommended further revision of the term "rubber" and also recommended a definition of the term "elastomer." These were considered by Subcommittee VIII and Committee D-11 at their meetings in February, 1961. These definitions have again been submitted to letter ballot of Committee D-11.

The title of Subcommittee XXII has been changed from "Cellular Rubber" to "Flexible Cellular Materials."

Subcommittee XIV on Abrasion Tests for Rubber Products and Subcommittee XVII on Hardness, Set, and Creep have been discharged and the work on these subjects assigned to Subcommittee X on Physical Testing of Rubber Products.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] *Materials Research & Standards*, Vol. 1, No. 4, April, 1961, p. 280.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1960 Annual Meeting, Committee D-11 presented to the Society through the Administrative Committee on Standards, the following recommendations which were accepted on the dates indicated:

New Tentative Method of Test for:

Specific Gravity of Rubber Chemicals (D 1817 - 61 T) (Accepted March 29, 1961).

Revision of Tentative Method of:

Tension Testing of Vulcanized Rubber (D 412 - 51 T) (Accepted March 1, 1961).

Revision of Tentative Methods for Chemical Analysis of:

Natural Rubber (D 1278 - 58 T) (Accepted March 1, 1961).

Synthetic Elastomers (Solid Styrene-Butadiene Copolymers) (D 1416 - 58 T) (Accepted Dec. 20, 1960).

Revision of Tentative Specifications and Methods of Test for:

Flexible Foams Made from Polymers or Copolymers of Vinyl Chloride (D 1565 - 59 T) (Accepted Dec. 20, 1960).

Revision of Tentative Recommended Practices for:

Description of Types of Styrene-Butadiene Rubbers (SBR) and Butadiene Rubbers (BR) (D 1419 - 59 T) (Two revisions, one accepted Dec. 20, 1960 and the second on March 1, 1961).

Description of Types of Styrene-Butadiene Rubber (SBR) and Butadiene Rubber (BR) Latices (D 1420 - 59 T) (Accepted Dec. 20, 1960).

Revision of Tentative Definitions of:

Terms Relating to Rubber and Rubber-like Materials (D 1566 - 58 T). (Accepted Sept. 29, 1960).

The revisions of D 1416, D 1419, D 1420, and D 1565 appear in the 1960 Supplement to Book of ASTM Standards, Part 9. The other items are available as separate reprints.

NEW TENTATIVES

The committee recommends for publication as tentative, the following specifications and methods of test as appended hereto:[2]

Tentative Specifications for:

Rubber Rings for Asbestos-Cement Pipe.

Tentative Methods of Test for:

Elevated Temperature Aging Using a Tubular Oven,

Adhesion of Vulcanized Rubber to Single-Strand Wire,

Free 2-Mercaptobenzothiazole in Benzothiazyl Disulfide Rubber Vulcanization Accelerator and

Total 2-Mercaptobenzothiazole in Commercial Benzothiazyl Disulfide Rubber Vulcanization Accelerator.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Methods for Chemical Analysis of Rubber Products (D 297 - 60 T):[3]

Revise as appended hereto.[4]

Tentative Specifications for Elastomer Compounds for Automotive Applications (D 735 - 60 T).[3]

Table V.—At the request of the SAE-ASTM Technical Committee on Automotive Rubber, it is recommended that Table V be revised as shown in the accompanying Table I.[5] Several new grades have been added, some revised, and others omitted.

Tentative Method of Test for Compressibility and Recovery of Gasket Materials (D 1147 - 59 T).[6]

Section 2(c).—Add a second sentence to read: "Readings shall be estimated to the nearest 0.0001 in."

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[2] The new tentatives appear in the 1961 Book of ASTM Standards, Part 11.

[3] 1960 Supplement to Book of ASTM Standards, Part 9.

[4] The revised method appears in the 1961 Book of ASTM Standards, Part 11.

[5] Not included here; see revised specifications, 1961 Book of ASTM Standards, Part 11.

[6] 1959 Supplement to Book of ASTM Standards, Part 9.

Table I.—In the column headed "Conditioning Procedure" change the description for Procedures F, B and G as follows:

Procedures F and B.—Add at the beginning the words "At least."

Procedure G.—In line 4, add the words "at least" before "by."

Change the last column to read as follows by the addition of the information on psi:

Procedure	Total Load (Sum of Major Load and Pre-Load) lb	psi
A	250	5000
H	50	1000
F	100	100
B	80	400
G	50	1000

Tentative Methods for Chemical Analysis of Synthetic Elastomers (Solid Styrene - Butadiene Copolymers) (D 1416 - 60 T):[3]

New Procedure.—Add as Sections 87 to 96 a new procedure for the visual measurement of the color of light-colored rubbers as appended hereto.[7]

Tentative Recommended Practice for Nomenclature for Synthetic Elastomers and Latices (D 1418 - 58 T):[8]

Section 4.—Add a new Paragraph (*c*) to read as follows:

(*c*) The "M" family includes elastomers having a saturation chain of the polymethylene type. The following classification shall be used.

IM—Polyisobutene.
EPM—Ethylene-propylene copolymer.
CSM—Chloro-sulfonyl-polyethylene.
CFM—Polychloro-trifluoro-ethylene.
FPM—Vinylidine fluoride and hexafluoropropylene copolymer.
ACM—Ethyl or other acrylate and 2-chloroethyl vinyl ether copolymer.
ANM—Ethyl or other acrylate and acrylonitrile copolymer.

Tentative Recommended Practice for Description of Types of Styrene-Butadiene Rubbers (SBR) and Butadiene Rubbers (BR) (D 1419 - 60a T):[3]

Section 2(d).—Add the following note as a means of listing ASTM test methods for testing several properties of the oils listed in Table II:

NOTE 2.—The oil types described in Table II may be tested by the following methods:

	Code Number Assigned			
	1024	1511	1610	1715
Type	1000	1500	1600	...
Nominal Temperature, deg Fahr	122	43	43	43
Activator	...	FRA	FRA	FRA
Shortstop	ND	ND	ND	ND
Antioxidant	NST	NST	ST	NST
Catalyst	P	OHP	OHP	OHP
Emulsifier	FA	FA	RA	FA
Nominal Bound Styrene, per cent	25	23.5	23.5	23.5
Nominal Conversion, per cent	68	60	60	60
Nominal Mooney Viscosity, ML 1 + 4 (212 F)—Polymer	30	40	...	45
Nominal Mooney Viscosity ML 1 + 4 (212 F)—Compound	35	70	57	...
Coagulation	ALUM	SA	ACID	GA
Carbon Black Type	...	...	ISAF	...
Carbon Black, PHR	...	...	52	...
Oil Type	...	...	HI-AR	NAPH
Oil, PHR	...	...	10	50
Finishing	normal	normal	normal	SP

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[7] The revised methods appear in the 1961 Book of ASTM Standards, Part 11.

[8] 1958 Book of ASTM Standards, Part 9.

Test Required	ASTM Method[a]
Viscosity	D 88 or D 445
Flash Point	D 92
Pour and Cloud Point	D 97
Aniline Point	D 611
Volatile Matter	D 972
Neutralization Value	D 974
Specific Gravity	D 1298
Color	D 1500

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[a] These designations refer to the following methods of the American Society for Testing Materials:

- D 88, Test for Saybolt Viscosity[8]
- D 92, Test for Flash and Fire Points by Cleveland Open Cup[4]
- D 97, Test for Cloud and Pour Points[8]
- D 445, Test for Kinematic Viscosity[3]
- D 611, Test for Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents[8]
- D 972, Test for Evaporation Loss of Lubricating Greases and Oils[8]
- D 975, Test for Neutralization Number by Color-Indicator Titration[8]
- D 1298, Test for Specific Gravity of Petroleum Liquids, Hydrometer Method[8]
- D 1500, Test for ASTM Color of Petroleum Products (ASTM Color Scale)[8]

Table II.—Add four new SBR polymers, the code numbers for which have been assigned since January, 1961, as shown at the bottom of p. 525.

Tentative Specifications for Carbon Blacks Used in Rubber Products (D 1765 - 60 T)[3] (Jointly with Committee D - 24):

Table II.—Experience indicates that a revision in the stress at 300 per cent elongation values is needed for GPF Black as follows:

Type	Time at 293 F, min	Stress at 300 per cent Elongation, psi	
		Min	Max
GPF	15	−400	+100
	30	−525	−25

ADOPTION OF TENTATIVES AS STANDARD WITH REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

Tentative Method of Measuring Low-Temperature Stiffening of Rubber and Rubber-Like Materials by Means of a Torsional Wire Apparatus (D 1053 - 58 T):[8]

Section 2(c).—Add as the second sentence, "The color codes for these wires are black, yellow, and white, respectively."

Section 4.—Change the second sentence to read, "The standard thickness of the specimens shall be the thickness of the material undergoing test but shall be within the limits of 0.085 ± 0.025 in."

Add the following to the end of this section:

> Values of thickness other than standard may be used provided it can be shown that they give equivalent results for the material being tested. When specimens taken from the finished article are not of standard thickness, it should be permissible, upon agreement between the manufacturer and the purchaser, to use a standard-size specimen, taken from a certified press-cured sheet of the same compound.

Tentative Method of Test for Viscosity and Curing Characteristics of Rubber by the Shearing Disk Viscometer (D 1646 - 59 T):[6]

Note 4.—Change the third sentence to read as follows to provide for a description of the type of cellophane and for the use of Mylar as films suitable for use with sticky materials:

> When testing low-viscosity or sticky materials, it is permissible to insert between the specimen and the die cavity a layer of untreated non-heat sealing, non-moistureproof cellophane (DuPont 600 PD/001 or Avisco 300P), a polyester film (Mylar Type A-100), or similar material approximately 0.001 in. thick.

Add the following as the fourth sentence: "Caution must be exercised in selecting this material to be sure there is no reaction between the test specimen and the film."

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods of Test for:

Abrasion Resistance of Rubber Soles and Heels (**D 1630 - 59 T**).[6]

Tentative Methods of Testing:

Adhesives for Brake Linings and Other Friction Materials (**D 1205 - 59 T**).[6]

Tentative Method for:

Strain Testing of Vulcanized Rubber (**D 1456 - 57 T**).[8]

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revision of the following standards as indicated, and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

Standard Method of Test for Compression Set of Vulcanized Rubber (D 395 - 55):[8]

Section 5.—Change to read as follows.[9]

Sections 9 and 12.—Add the following as a new Paragraph (*e*) to each of these sections: "(*e*) The type of test specimen used together with the time and temperature of cure."

Section 11(*b*).—Change the last sentence to read, "The percentage of compression employed shall be 25 per cent for all hardnesses and the thickness of the spacer bar shall be 0.375 ± 0.001 in."

Section 12.—Delete Footnote 3.

Standard Method of Test for Resistance to Surface Cracking of Stretched Rubber Compounds (D 518 - 60):[8]

Sections 4 and 8 specify that the test specimens shall be cut longitudinally from standard laboratory test slabs. Section 12 does not state the direction. Since the standard test slab described in Section 13 (*b*) of the Tentative Methods of Sample Preparation for Physical Testing of Rubber Products (ASTM D 15 - 59 T) has dimensions 6 by 6 in. there is no longitudinal direction. Therefore, the committee recommends the following revision:

Section 4(*a*).—Change to read as follows:

> (*c*) The test specimens shall be rectangular strips 1 in. in width by 6 in. in length cut from standard laboratory test slabs having a thickness of 0.075 in. minimum and 0.100 in. maximum so that the grain will be in the lengthwise direction of the specimen.

Section 8(*a*).—Change to read as follows:

> 8. (*a*) The test specimens shall be rectangular strips 1 in. in width by 3¾ in. in length cut from standard laboratory test slabs having a thickness of 0.075 in. minimum and 0.100 in. maximum so that the grain will be in the lengthwise direction of the specimen.

Section 12(*a*).—Change to read as follows:

> 12. (*a*) The test specimens shall be die-cut tapered strips having outside dimensions as shown in Fig. 4, cut from standard laboratory test slabs having a thickness of 0.075 in. minimum and 0.100 in. maximum so that the grain will be in the lengthwise direction of the specimen.

Standard Method of Test for Accelerated Aging of Vulcanized Rubber by the Oxygen Pressure Method (D 572 - 53):[8]

Copper contamination and its contribution to erroneous results is still a problem. Therefore, the committee recommends the following revision:

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[9] Not included here, see the revised method, 1961 Book of Standards, Part 11.

Section 4(*h*).—Change to read, "(*h*) No copper or brass parts shall be exposed to the atmosphere, nor used in the pressure chamber and tubing or valves leading to it."

Standard Method of Test for Physical State of Cure of Vulcanized Rubber, (T-50 Test) (D 599 – 55):[8]

Section 1.—Change the fourth sentence to read, "Its use is limited primarily to natural rubber compounds. . . ."

Standard Method of Test for Weather Resistance Exposure of Automotive Rubber Compounds (D 1171 – 59):[8]

Section 2.—Add a new Paragraph (*c*) to read as follows:

(*c*) *Test Chamber.*—An ozone chamber as described in the Method of Test for Accelerated Ozone Cracking of Vulcanized Rubber (ASTM Designation: D 1149).

Section 4(*b*).—In Paragraph (*1*), change the rest period from "48 hr" to read "70 to 72 hr." Delete the words "a minimum of." Add to the end of the paragraph "Care must be taken to prevent looped parts of the specimen from touching the hands or anything else after mounting, until the test is completed."

Change Paragraph (*2*) to read as follows:

> (*2*) *Angle and Direction of Exposure.*—Specimens exposed outdoors shall be so inclined that the portion of the loop diametrically opposite the tied ends shall face south at an angle of 45 deg with the horizontal as shown in Fig. 2. Or they may be exposed in a cabinet in which the ozone concentration is controlled as described in the Method of Test for Accelerated Ozone Cracking of Vulcanized Rubber (ASTM Designation : D 1149). The position of the mounted specimens exposed in the ozone test chamber shall be such that there is complete circulation of air around the specimen, and the specimens do not touch anything during the necessary handling.

Change Paragraph (*3*) to read as follows:

> (*3*) *Test Conditions:*
>
> (A) *Outdoor Exposure.*—The type, time and temperature, and other essential conditions of exposure shall be agreed upon by the purchaser and the seller.
>
> (B) *Ozone Chamber Exposure.*—The test shall be run for 72 hr at 100 ± 2 F at an ozone concentration of 50 ± 5 parts per 100 million parts of air per volume, unless otherwise specified by agreement between the purchaser and the seller. The specimens may be removed from the test chamber after 24, 48, and 72 hr exposure, for rating.

Section 4(*c*).—Delete the words "as follows" and the table of rating number and description. Add as the second sentence "If the size of the cracks under 2X magnification is less than those shown on one photograph but greater than those shown on the next lower rating picture, it shall be given the lower rating."

Figure 3.—Change as shown in the accompanying Fig. 1.[9]

Standard Methods of Testing Synthetic Rubber Latices (Styrene-Butadiene Copolymers) (D 1417 – 59):[6]

The following two revisions are recommended to clarify the procedures for surface tension and coagulum content:

Section 8(*a*).—Change to read as follows:

> 8. (*a*) *Surface Tension.*—The surface tension of styrene-butadiene rubber latex shall be determined on the total solids of 40 per cent. If the viscosity is below 200 cp on No. 1 spindle at 20 rpm, the latex can be tested with solids as received with little loss in accuracy.

Section 10(*c*).—Add as the third sentence, "Clean the stainless steel screen by burning."

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the remaining tentatives under its jurisdiction. Many of them are under consideration for revision.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[10]

This report has been submitted to letter ballot of the committee which consists of 272 members; 181 members returned their ballots, of whom 133 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

S. COLLIER,
Chairman.

JOHN J. ALLEN,
Secretary.

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[10] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE D-12
ON
SOAPS AND OTHER DETERGENTS*

Committee D-12 on Soaps and Other Detergents held one meeting during the year: on March 6 and 7, 1961, in New York, N. Y. Four papers were presented at the meetings: "Foam Measurement by a Dynamic Method" by H. E. Reich, J. T. Patton, and C. V. Francis; "Photometric Measurement of Filming and Water Spotting in Dishwashing" by A. L. Kimmel, D. O. Darby, and H. M. Gadberry; "Low Foam Nonionic Surfactants" by J. Dupre, R. E. Wolfrom, and D. B. Fordyce; "Notes on the Mechanism of Soil Removal" by J. C. Harris.

The committee consists of 122 members, of whom 62 are classified as producers, 34 as consumers, and 26 as general interest members, with 7 consulting members.

Accepted with regret, and with full appreciation of his accomplishments, was the resignation of J. L. Darragh, former chairman of Subcommittee T-5 on Physical Testing. Appointed as his replacement was L. E. Weeks.

Reapproval of Standards

The committee recommends the reapproval of the following standards, most of which have stood for six or more years without revision:

Standard Specifications for:

Milled Toilet Soap (**D 455 - 55**),
Caustic Soda (Anhydrous) (**D 456 - 39**),
Modified Soda (Sesquicarbonate Type) (**D 457 - 39**),
White Floating Toilet Soap (**D 499 - 48**),
Built Soap, Powdered (**D 533 - 44**),
Alkaline Soap Powder (**D 534 - 42**),
Sodium Metasilicate (**D 537 - 57**),
Trisodium Phosphate (**D 538 - 60**),
Sodium Sesquisilicate (**D 594 - 41**),
Tetrasodium Pyrophosphate (Anhydrous) (**D 595 - 45**),
Sodium Bicarbonate (**D 928 - 52**), and
Borax (**D 929 - 50**).

Standard Methods of:

Chemical Analysis of Sulfonated and Sulfated Oils (**D 500 - 55**),
Test for Particle Size of Soaps and Other Detergents (**D 502 - 39**),
Total Immersion Corrosion Test of Water-Soluble Aluminum Cleaners (**D 930 - 49**), and
Test for Foaming Properties of Surface-Active Agents (**D 1173 - 53**).

The committee recommends the reapproval of the following standards with editorial changes as indicated:

Standard Specifications for Chip Soap (D 496 - 51):

Section 2.—After the word "fats" in the first sentence, add the words "or fatty acids, or both."

Standard Specifications for Ordinary Laundry Bar Soap (D 497 - 52):

Section 2(a).—Change as indicated above for Specifications D 496.

Standard Specifications for Powdered Soap (Nonalkaline Soap Powder (D 498 - 51):

Section 2.—Change as indicated above for Specifications D 496.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

Standard Specifications for Salt-Water Soap (D 593 – 42):

Title.—Add as a subtitle "Coconut Oil Type."

Section 2.—After the work "thereof" in the first sentence, add the words "or of their fatty acids."

Standard Specifications for Olive Oil Chip Soap (Type A, Straight; Type B, Blended) (D 630 – 42):

Section 2(b).—Change as indicated above for Specifications D 496.

Standard Specifications for Compound Chip Soap (D 690 – 48):

Section 2.—Change "made from soda, fats, and rosin" to read "made from soda, fats, or fatty acids or both, and rosin."

Standard Specifications for Compound Powdered Soap (Granulated, with Rosin) (D 691 – 44):

Section 2.—Change as indicated above for Specifications D 690.

Standard Specifications for Chip or Granular Soap for Low-Temperature Washing (Low and Medium Titer) (D 1111 – 53):

Section 2.—Change as indicated above for Specifications D 496.

Standard Specifications for Solid Soap for Low-Temperature Washing (Low and Medium Titer) (D 1112 – 53):

Section 2.—Change as indicated above for Specifications D 496.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Methods of:

Testing Detergent Cleaners for Evaluation of Corrosive Effects on Certain Porcelain Enamels (**D 1567 - 58 T**),

Testing Detergent Alkylate (**D 1569 - 59 T**), and

Test for Synthetic Anionic Active Ingredient in Detergents by Cationic Titration Procedure (**D 1681 – 59 T**).

Tentative Definitions of Terms Relating to:

Soaps and Other Detergents (**D 459 - 58 T**).

The committee recommends the continuation without revision of the following tentatives, with editorial changes as indicated:

Tentative Methods for Sampling and Analysis of Sodium Triphosphate (D 501 – 59 T):

Section 8(c).—Correct "20 volumes of water" to read "1 volume of water."

Section 43.—Add after the second sentence "The relative quantities of Forms I and II are determined by the temperature rise test as herein described."

Tentative Specifications for Liquid Toilet Soap (D 799 – 60 T):

Section 2(a).—Add "or both" after "distilled fatty acids."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[1]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee S-2 on Specifications for Soaps and Synthetic Detergents (W. H. Joy, chairman):

Task Group 1 on Synthetic Detergents (R. E. Hauber, chairman).—Reorganization of the group is under way.

Task Group 2 on Scouring Powders (C. A. Gerardi, chairman) presented a rough draft of a specification for scouring powders which will be refined during the year.

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[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Task Group 3 on Synthetic and Combination Bars (R. M. Kelley, chairman) will develop specifications for synthetic and synthetic-soap combination bars.

Task Group 4 on Building Cleaning Materials (L. V. Hassel, chairman) will develop areas of interest and possible specifications.

Subcommittee T-2 on Analysis of Soaps and Synthetic Detergents (J. C. Harris, chairman):

Task Group 1 on Classification of Surfactants (H. C. Speel, chairman) is evaluating the proposed ISO system for its application to punch card or IBM retrieval, and for analytical guidance.

Task Group 3 on Bibliographical Abstracts of Analytical Methods for Surfactants (J. C. Harris, chairman) developed the 1959 Supplement to the Bibliographical Abstracts of Methods for Analysis of Synthetic Detergents (*STP No. 150-C*).

Task Group 4 on Separation of Active Ingredient (R. M. Kelley, chairman) will investigate the ethanol separation, followed by an acetone-ether step, so that the active ingredient may be used for quantitative separation, qualitative identification, or infrared examination.

Task Group 5 on Infrared Determination of Surfactants (E. A. Setzkorn, chairman) has examined a number of surfactants for functional group estimation and will expand these activities in a further interlaboratory series.

Task Group 8 on Determination of Carboxymethyl Cellulose (J. L. Darragh, chairman), upon investigating a shorter procedure, found some discrepancies at lower concentration levels. Upon resolution of these differences, a further cooperative test will be initiated.

Task Group 16 on Qualitative Method for Identification of Surfactants and Organic Builders (L. E. Weeks, chairman) tested a procedure for single component materials with complete success. The next trials will encompass multicomponent mixtures.

Task Group 20 on Total Active Ingredient (C. A. Cohen, chairman) prefers the two-phase isopropanol - sodium carbonate separation procedure for its ability to minimize the presence of electrolytes in the separated active ingredient. Collaborative tests between this Task Group and Task Group 4 are to be initiated.

Task Group 21 on Detergent Alkylate (J. C. Harris, chairman):

Subgroup A on Distillation Range (J. L. Darragh, chairman) continues its liaison with other committees under whose sponsorship various distillation methods are under scrutiny.

Subgroup B on Molecular Weight (C. A. Cohen, chairman) reports that the only precise method is that by mass spectroscopy. In the absence of such equipment a cryoscopic procedure is recommended, with the notation that the refractive index - viscosity - density procedure did not perform successfully.

Subgroup C on Acid Wash Color (R. C. Stillman, chairman) is awaiting the results of the revision of the procedure by Committee D-16 on Industrial Aromatic Hydrocarbons before further evaluation for detergent alkylate.

Task Group 22 on Estimation of Ethylenediamine Sodium Tetraacetate (EDTA) and Other Sequestrants (E. W. Blank, chairman) has circulated a test method for sequestrants other than EDTA and expects to perform a cooperative test during the year.

Task Group 25 on Alkylaryl Sulfonates (R. C. Stillman, chairman) has a test method under cooperative investigation. Quantitative separation and identification of the lower alkylaryl sulfonates is expected.

Task Group 26 on Nonionic Surfactants

(M. J. Rosen, chairman) is attempting to secure a nonionic of assured purity (most of these represent a molecular weight distribution). Various procedures for identification and analysis of the hydrophobe and hydrophil portions of the molecule are under study.

Subcommittee T-5 on Physical Testing (J. L. Darragh, chairman):

Task Group 1 on Measurement of Soil Redeposition (R. C. Davis, chairman) is considering clay as a model soil.

Task Group 3 on Measurement of Reflectance of Test Fabrics (R. L. Liss, chairman) has developed supplies of uniform materials and will circulate kits and NBS standard plaques for cooperative measurement.

Task Group 5 on Brightener Evaluation (M. S. Furry, chairman) is attempting to develop methods for determining buildup, substantivity, and stability to light and to bleaching agent of brightener-treated fabrics.

Task Group 9 on Methods of Determination of Cloud Points of Detergents (F. E. Woodward, chairman) plans to develop and evaluate a procedure during the coming year.

Task Group 10 on Methods for Evaluation of Detergents in Dishwashing (R. E. Johnson, chairman) compiled a series of tests used in various laboratories which will be studied to develop those to be evaluated cooperatively.

Task Group 11 on Studies of Primary Deposition of Soil (A. M. Schwartz, chairman) continues a card file of selected references. Expansion of interest to soil-resistant treatments may prove within the scope of task group activities.

Respectfully submitted on behalf of of the committee,

J. C. HARRIS,
Chairman.

H. R. SUTER,
Secretary.

REPORT OF COMMITTEE D-13
ON
TEXTILE MATERIALS*

Committee D-13 on Textile Materials held two meetings during the year: in New York, N. Y., on Oct. 18 to 21, 1960, and Feb. 28 to March 3, 1961. At each meeting over 60 subcommittees and task groups met to discuss revisions of current standards and development of new methods.

At the October meeting, Messrs. K. L. Hertel, Burt Johnson, and A. G. Scroggie presented three papers at a general session summarizing the actions and activities of the meetings of ISO/TC 38 on Textiles held in London, England in May, 1960. The American delegation of 17 was headed by Mr. Hertel. His leadership and the hard and outstanding work of Messrs. Scroggie and Johnson, and members of Subcommittees A-1, B-2, B-6, and B-8, are gratefully acknowledged.

The demand for reprints from the ASTM Bulletin of "Introduction of the Tex System for Designation of Yarn Number"[1] continued throughout the year, especially from foreign countries. These countries are looking to the United States for leadership in introducing the tex system in all segments of the textile industry. This challenge to show foresight and leadership should be accepted unhesitatingly. The opportunity to obtain the tremendous benefits of the tex system and to pass these benefits on to future generations of the textile industry of all nations must be seized promptly and courageously. In addition to the huge economic values that will accrue in all future years by adopting the tex system (to end the utter confusion of the great multiplicity of current systems), there are the inestimable and intangible values of showing to all the world how established traditions and practices can and must give way to modern, unified, and harmonious practices, concepts, and mutual understanding. To help attain this goal many members of Committee D-13 participated in the technical program of the American Association for Textile Technology held in New York City on March 1, 1961. This program was sponsored jointly by AATT and Subcommittee B-8 of ASTM Committee D-13. A. G. Scroggie outlined in detail the present multiplicity of yarn numbering systems with their innumerable disadvantages, and clearly indicated the many advantages of the tex system. This excellent paper is published in *Modern Textiles Magazine*, Vol. 42, p. 61, April, 1961. It is recommended very highly and sincerely for careful study.

The Task Group on Yarn Appearance Standards, of Subcommittee B-8, met at both meetings of Committee D-13. At the March meeting approximately 800 photographs were carefully compared with the master standards and some

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] ASTM Bulletin, No. 247, July, 1960, p. 48.

700 were judged acceptable for standards and the remainder, which were found unacceptable, were destroyed. Since the establishment of these yarn appearance standards by this task group some 40,000 photographs have been compared with the master standards. The Cotton Yarn Appearance Standards consist of twenty $5\frac{1}{2}$ by 10-in. approved photographs representing five series of yarn numbers. Each series is divided into four Grades A to D mounted on a board $27\frac{1}{2}$ by 15 in. for yarn comparison purposes. To date some 7500 boards have been sold by the Society to users all over the world. The dedicated work of this task group is worthy of special commendation.

At the March meeting the Harold DeWitt Smith Memorial Medal for 1961 was presented to Lyman E. Fourt, Associate Director of Harris Research Laboratories, in recognition of his outstanding contributions to textile science.

Herbert J. Ball, Honorary Chairman of Committee D-13, chairman for twenty years, and also a Past President of the Society, was elected an Honorary Member of Committee D-13 in recognition of his many years of outstanding and dedicated service to the work of the committee.

The following appointments have been made during the year: S. C. Mayne, Jr., chairman of Subcommittee B-3 on Atmospheric Conditions and Regain, to replace E. N. Ditton; N. L. Enrick, chairman, and W. C. Stuckey, Jr., secretary of Subcommittee B-5 on Sampling, Presentation, and Interpretation of Data; Braham Norwick, chairman to replace A. G. Scroggie, and H. A. Thompson, secretary to replace B. Norwick of Subcommittee B-6 Coordinating Committee for ISO and Foreign Textile Standards; E. A. McMasters, secretary of Section I on Automotive Fabrics of Subcommittee B-9, to replace H. R. Walker; Charles Bradford, acting chairman of new Section II on Heavy Industrial Fabrics, of Subcommittee B-9; S. J. Hayes, chairman of Subcommittee C-5 on Significance of Tests; A. G. Scroggie, chairman of new Subcommittee C-6 on Editorial Review and Policy.

RECOMMENDATIONS AFFECTING STANDARDS

The committee is presenting a number of recommendations relating to standards which are listed in detail in the Appendix.[2]

All other standards and tentatives not specifically referred to are being studied by the respective subcommittees and it is recommended that they be continued in their present status.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

This report has been submitted to letter ballot of the committee, which consists of 401 voting members; 200 members returned their ballots, of whom 172 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. F. SCHIEFER,
Chairman.

H. A. EHRMAN,
Secretary.

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[2] See p. 536.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS ON TEXTILE MATERIALS

In this Appendix proposed recommendations are given affecting certain standards and tentatives covering textile materials. The current standards and tentatives in question appear in their present form in the 1958 Book of ASTM Standards, Part 10, and its 1959 and 1960 Supplements.

REVISION OF PROPOSED METHOD

The committee recommends the revision of the Proposed Method of Test for Length and Length Distribution of Cotton Fibers by the Short Method Array, as appended hereto.[1] This method was formerly published as information in the compilation of "ASTM Standards on Textile Materials," Nov., 1960.

NEW STANDARD

The committee recommends for publication as a separate standard the Standard Method of Test for Diameter of Wool and Mohair Fibers by Microprojection as appended hereto,[2] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the method may be referred to letter ballot of the Society. This method includes those parts of the Standard Specifications and Methods D 419 - 58, D 472 - 56, and D 1381 - 57 which apply to methods of test.

NEW TENTATIVES

The committee recommends for publication as tentatives the following methods, definitions, and recommended practice as appended hereto[2] or as otherwise indicated:

Tentative Methods of Test for:

Dimensional Change in Woven and Knitted Textiles (Excluding Wool). (This tentative is a contemplated revision and consolidation of the following five methods: D 416 - 54 T, D 437 - 36, D 1470 - 59 T, D 1487 - 57 T, and D 1773 - 60 T),

Estimating Maturity and Weight per Unit Length of Cotton Fibers by the Causticaire Method (Adoption as tentative of Appendix XII, Proposed Method of Test for Estimating Maturity and Weight per Unit Length of Cotton Fibers by the Causticaire Method, with editorial changes),[3]

Estimation of Effective Gage Lengths in Single Fibers Testing,

Moisture Content and Moisture Regain of Lint and Seed Cotton (Oven Method),

pH of Aqueous Extracts of Wool and Similar Animal Fibers,

Rapid Estimation of Staple Length of Wool Top,

Yarn Number (Skein Method). (This method replaces parts of the following methods: D 180 - 57 T, D 1285 - 58 T, and D 1380 - 57 T),

Yarn Severance in Woven Fabrics,

Colorfastness of Zipper Stringers to Dry Cleaning,

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[1] The revised method appears in the compilation of "ASTM Standards on Textile Materials," Nov., 1961.

[2] The new standard and tentatives appear in the 1961 Book of ASTM Standards, Part 10.

[3] Compilation of "ASTM Standards on Textile Materials," November, 1960.

Colorfastness of Zipper Tapes to Crocking,
Colorfastness of Zipper Tapes to Perspiration,
Colorfastness of Zippers to Laundering,
Colorfastness of Zippers to Light,
Durability of Finish of Zippers to Dry Cleaning,
Durability of Finish of Zippers to Laundering,
Measuring Zippers,
Operability of Zippers,
Resistance of Finish of Zippers to Dry Abrasion,
Resistance of Zippers to Salt Fog, and
Strength Tests of Zippers.

Tentative Definitions of Terms Relating to:

Zippers.

Tentative Listing of:

Commercial Moisture Regains for Textile Fibers.

Tentative Recommended Practice for:

Establishment of Standard Moisture Content for Wool and Its Products, as appended hereto.[2] (This replaces Appendix IX, Recommendations for the Establishment of Standard Moisture Content for Wool and Its Products).[3]

REVISION OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Methods of Testing and Tolerance for Cotton Yarns (D 180 - 57 T):

Section 10.—Replace Section 10 with a reference to the Tentative Method of Test for Yarn Number of Textile Yarns by Skein Method (ASTM Designation: D 1907).

Section 14 (a).—Replace Item (*1*) with a reference to the Tentative Method of Test for Yarn Number of Textile Yarns by Skein Method (ASTM Designation: D 1907).

Table I.—Replace the first line of this table with a reference to the Tentative Method of Test for Yarn Number of Textile Yarns by Skein Method (ASTM Designation: D 1907).

Tentative Methods for Identification of Fibers in Textiles (D 276 - 60 T):

Section 1.—Add in the proper alphabetical order the following items: Acrilan 16®, Arnel®, Dynel 60®, Dynel 97®, Lycra®, Orlon 21®, Orlon 39®, Polyporphylene, Rilsan®, Rovana®, Teflon®, Verel®, Vinal, Vycron®, and Vyrene®.

Delete the following fibers: Velon® and Vinyon HH®.

Change the last sentence to read as follows: "Although Fibravyl, Merinova, Perlon, Rhovyl, Rilsan, and Rovana are fibers from other countries, they are included because they may be encountered in the United States."

Section 2.—In the fifth sentence, change "Appendix" to read "Appendix I." Also, add as the last sentence "Infrared spectra included in Appendix II are not influenced by shape factors, but changes in manufacturing processes may alter the spectra, so that current samples should be checked in case of doubt."

Section 3.—Add new Paragraphs (*w*) and (*x*) to read as follows: "(*w*) *Potassium Bromide*, reagent grade for infrared examination," and "(*x*) Chloroform, reagent grade for infrared examination."

Section 4.—Add a new Paragraph (*c*) to read: "(*c*) *Infrared Spectrophotometer*, double-beam instrument reading from 2.5 to 15.0 μ."

Section 6.—Add a new Paragraph (*c*) to read as follows:

> (*c*) *Preparation for Infrared Examination.*—Normally dyestuffs and finishes will not interfere with determination of the qualitative nature of the fiber substance by infrared. If fiber mixtures are present, these must be separated prior to testing for spectra either by the density gradient column or by selective solvents such as noted in Table VI of Appendix II. The fibers are ground with potassium bromide and hydraulically pressed to form a disk, or made into a film with suitable solvents as recommended.

Section 8(d).—In the list of fibers, delete "Velon®" and "Vinyon HH®" and add in the proper alphabetical order the following: Acrilan 16®, Dynel 60®, Dynel 97®, Orlon 21®, Orlon 39®, Rilsan®, and Lycra®.

TABLE I.—THE CROSS-SECTION, DENSITY, AND SOLUBILITY OF FIBERS IN DIFFERENT SOLVENTS.
(Addition to Table V of Methods D 276)

Fibers	Cross-Section	Density Range, g per ml	Nitric Acid	Acetone	HCl (1:1)	H_2SO_4, 70 per cent	NaClO	Methylene Chloride	NaOH, 45 per cent	*nn*'Dimethyl Formamide	*m*-Cresol
Acrilan 16®............	round to bean	1.17	+	−	−	−	−	−	− (Browns)	+	− (Fused)
Creslan®...............	round	1.18	+	−	−	−	−	−	− (Reddens)	+	− (Partial)
Dynel 60®.............	ribbon	1.28 to 1.31	− (fused)	+	−	−	−	−	− (Fused)	+	+ (Boil)
Dynel 97®.............	round	1.28 to 1.31	− (fused)	+	−	−	−	−	− (Fused)	+	+ (Boil)
Kodel®................	round	1.21 to 1.24	−	−	−	−	−	−	+	−	+ (Boil)
Lycra®................	dog bone	1.0	partial	−	−	+	−	−	−	−	+ (Boil)
Orlon 21®.............	T-shaped	1.17	+	−	−	−	−	−	− (Reddens)	−	−
Rilsan®...............	round	...	− (fused)	−	−	−	−	−	−	−	+

Table V.—Delete "Velon®" and all corresponding requirements. Also, add in proper alphabetical order the fibers listed in the accompanying Table I.

Table VI.—Replace with the accompanying Table II.[4]

Table VII.—Delete "Velon®" and all corresponding requirements, and add in the proper alphabetical order the following:

Fiber	Refractive Indexes		Birefringence	Source
	ε	ω	ε − ω	
Dynel 60®.....	1.54	1.53	0.01	*k*
Dynel 97®.....	1.54	1.53	0.01	*k*

k = Union Carbide Chemicals Co.

New Table.—Add as a new Table VIII the accompanying Table III.[4]

New Section.—Add the following as a new Section 13:

13. *Procedure for Melting Point Determination.*—(*a*) *Scope.*—This method is intended for determining the melting point of thermoplastic fibers, thread, and yarn containing a single type of fiber.

(*b*) *Apparatus.*—An apparatus containing an electrically heated stage with controllable heat input, an armored thermometer inserted into a circular depressed stage, and auxiliaries such as a magnifying eyepiece and an illuminating lamp. The instrument should have a range of 100 to 300 C or more, and an accuracy within ±1 deg over the entire range. The instrument should contain a graduated powerstat dial for ease of operation and reproducibility of settings (Note).

NOTE.—One such unit is the Fisher-Johns Melting Point Apparatus.

(*c*) *Test Specimens.*—The specimen shall be 3 to 4 mg of material undergoing test.

(*d*) *Standardization.*—Standardize the apparatus with U. S. Pharmacopeia Reference Standards or other suitable means.

(*e*) *Procedure.*—If the approximate melting point of the specimen is not known before testing, determine it by a trial run. Place the specimen between two cover glasses and lay the entire unit on the hot stage and press gently but firmly into place. Raise the temperature of the stage rapidly to within 15 C of the expected melting point and thereafter at a rate of 3 to 4 C per min. At this time, apply a slight pressure on the upper microglass cover by pressing with a spatula or pick needle. Observe the specimen with the aid of the magnifying glass and take the melting point as the temperature at which the flow of the specimen is observed. At the observed melting point, read the thermometer to the nearest degree. In subsequent determinations immediately following the initial determination, cool the stage in each case to approximately 50 C below the expected melting point before the specimen is put in place for testing. Report the melting point as the average of two determinations on each unit-of-produce to the nearest 1 C.

Figure 23.—Replace with the accompanying Fig. 1.[4]

New Appendix.—Add as Appendix II the infrared spectra appended hereto.[4]

Tentative Methods of Test for Abrasion Resistance of Textile Fabrics (D 1175 - 55 T):

Title.—Revise to read: "Tentative Recommended Practice for Operating Machines for Testing Abrasion Resistance of Textile Fabrics."

Sections 29 to 34.—Change Procedure E to read as appended hereto.[4]

Tentative Method of Test for Average Fiber Diameter of Wool Tops, Card Sliver, and Scoured Wool by Micronaire Method (D 1282 - 57 T): Revise as appended hereto.[4]

Tentative Methods of Testing Tufted Pile Floor Coverings (D 1486 - 57 T):

Section 2.—Add to Paragraph (*b*) the following sentence: "Equilibrium shall be approached from the dry side (not moisture free)."

In the first sentence of Paragraph (*c*) replace "the standard atmosphere" by "the specified atmosphere." Delete the last sentence of this paragraph.

Section 4(d).—Change to read as follows:

(*d*) The three measurements shall consist of one measurement taken in the center of the sample and one measurement taken at a point six inches from each edge.

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[4] Not included here; see revised method, 1961 Book of ASTM Standards, Part 10.

Section 10.—Add a note to read as follows:

NOTE.—It should be pointed out that the net pile thickness as determined in Sections 8, 9, and 10 can be considered to be the "average thickness" of the sample only in the case of fabric with a uniform pile height. It cannot be interpreted in this manner for fabrics of variable pile height.

Section 15(a).—Change "10 by 10-in." to read "9 by 9-in."

Section 15(c).—Change "10 by 10-in. sample" to read "9 by 9-in. sample, approximately 81 sq in."

Section 16(b).—Change to read as follows:

(*b*) Where the specifications of the sample are known, the sample shall be a 9 by 9-in. square determined by counting a number of rows equal to nine times the number of rows per inch and a number of pile ends equal to nine times the face pitch.

Section 16(d).—Change to read: "(*d*) Carefully separate the various yarns according to their use in the fabric, that is pile, chain, stuffer, and filling."

Section 17.—Change Paragraph (*b*) to read as follows:

(*b*) *Backing.*—Weigh each of the various backing yarns to the nearest 0.01 oz, and take these weights as the conditioned weights of the chain, stuffer, and filling.

In Paragraph (*d*) insert after "backing" the word "yarns."

Section 19(a).—Change size of sample to "9 by 9-in."

Tentative Methods of Testing and Tolerances for Yarns Containing Wool (D 1285 – 58 T):

Section 18.—Replace Section 18 with a reference to the Tentative Method of Test for Yarn Number (Skein Method) (ASTM Designation: D 1907).

Tentative Methods of Testing Spun and Filament Yarns Made Wholly or in Part of Man-Made Organic Base Fibers (D 1380 – 57 T):

Scope.—Delete the references to Sections 5 to 17.

Section 3(a).—Replace Paragraph (*a*) with a reference to the Tentative Method of Test for Yarn Number (Skein Method) (ASTM Designation: D 1907).

Sections 5 to 17.—Replace Sections 5 to 17 with a reference to the Tentative Method of Test for Yarn Number (Skein Method) (ASTM Designation: D 1907).

Tentative Method of Test for Elastic Properties of Uncrimped Fibers (D 1774 – 60 T): Revise as appended hereto.[4]

Tentative Method of Conditioning Textiles and Textile Products for Testing (D 1776 – 60 T):

Section 3.—Change Paragraph (*a*) to read as follows:

(*a*) *Standard Atmosphere for Testing.*—Standard atmosphere for testing is defined as in Definitions of Terms Relating to Textile Materials (ASTM Designations: D 123).

Change Paragraph (*c*) to read as follows, retaining Note 2:

(*c*) *Standard Atmosphere for Preconditioning.*—Standard atmosphere for preconditioning is defined as an atmosphere having a relative humidity of 5 to 25 per cent and a temperature of not over 50 C (122 F) (Note 2).

Section 4(b)(1).—Change to read as follows:

(*1*) Cabinet, room, or other container equipped with apparatus for maintaining the standard atmosphere for preconditioning throughout the cabinet, room, or other container within the tolerance given in Section 3(*c*).

Section 5.—In the fourth line of Paragraphs (*a*) and (*b*) substitute the word "material" for the word "detail."

Section 6.—In the second line of Paragraph (*b*) substitute the word "material" for the word "detail."

In the fourth line of Paragraph (*e*) add the word "it" after the word "maintain."

TENTATIVE REVISION OF STANDARDS

The committee recommends tentative revisions as follows of the Standard Definitions of Terms Relating to Textile Materials (**D 123 – 60**):

Add the new tentative definitions for the terms "Wrinkle," "Crease," "Pressed-in-Crease," "Wrinkle Resistance," "Wrinkle Recovery," "Pressed-in-Crease Retentions," "Mussiness," "Minimum Care," "Pleat," and "Textile" to read as follows:

Wrinkle, *n.*—An undesirable fabric deformation, usually a sharp or rounded short fold.

NOTE.—The distinction between a wrinkle and an undesirable crease is not clear; generally, short and rounded irregular deformations are referred to as wrinkles while longer and sharper ones are called creases.

Crease, *n.*—A fabric deformation, usually a long sharp fold either desirable or undesirable, depending on the situation.

Pressed-in-Crease, *n.*—A sharp crease inserted intentionally in a fabric usually by application of pressure, heat and moisture.

Wrinkle Resistance, *n.*—That property of a fabric which enables it to resist the formation of wrinkles when subjected to a folding deformation.

NOTE 1.—The apparent wrinkle resistance of a specific fabric may vary depending upon conditions attending the deformation and recovery, e.g., on temperature, moisture content of the fabric, force, duration, and direction of deformation.

NOTE 2.—The wrinkle resistance of a fabric is a valuable concept, but is not easily measured quantitatively. It varies from "quite low" in many fabrics to "very high" in resilient fabrics. Formation of a wrinkle overcomes a fabric's wrinkle resistance, which may produce strains and store potential energy that can become evident as wrinkle recovery under suitable conditions.

NOTE 3.—"Crease Resistance" is a term commonly used in place of the preferred term "Wrinkle Resistance."

Wrinkle Recovery (Crease Recovery), *n.*—That property of a fabric which enables it to recover from folding deformations.

NOTE.—The extent of wrinkle recovery may be expressed in degrees or percentage, or subjectively. Because it is not practical to determine the time required for complete recovery, test results are usually based on an arbitrarily chosen time, such as one second, five minutes, twelve hours, etc.

Pressed-in-Crease Retention, *n.*—That property of a fabric which enables it to maintain an inserted crease.

NOTE.—Pressed-in crease retention can be measured by the relation of a crease in a subsequent state to the crease in the initial state, or subjectively. This property may be strongly dependent upon the conditions of use through which retention is desired, e.g., normal wear, washing, tumble drying, etc.

Mussiness, *n.*—Surface distortion in a fabric characterized by undesirable unevenness due to many minor deformations.

NOTE.—Mussiness is independent of the presence or absence of soil.

Minimum Care, *n.*—A term applied to fabrics, garments, and household textile articles which can be washed satisfactorily by normal home laundering and used or worn after light ironing.

NOTE.—By "light ironing" is meant no starching, no dampening, and a relatively small expenditure of physical effort.

Pleat, *n.*—Three layers of fabric involving two folds or reversals of direction; the back fold may be replaced by a seam.

NOTE.—Pleats may be either pressed to give sharp creases or left unpressed to give soft folds.

Textile, *n.*—Originally a woven fabric, now generally applied to:

(*1*) Staple fibers and filaments suitable for conversion to or use as yarns, or for the preparation of nonwoven fabrics,

(*2*) Yarns made from natural or manmade fibers,

(*3*) Fabrics and other manufactured products made from fibers as defined above and from yarns, and

(*4*) Garments and other articles fabricated wholly from one or more of the above elements, and articles made principally from the above when the products retain the characteristic flexibility and drape of the original fabrics.

NOTE.—Typical examples (but not a complete listing) of material included in the above definitions are:

(*1*) Natural fibers such as cotton, wool, sisal, and asbestos, also silk filaments, after these materials have been subjected to their first preparatory process, and man-made fibers such as rayon, nylon, glass, and metals in the form of spinnable fibers, monofilaments, multifilament yarns or tows.

(*2*) Yarn intermediates such as sliver, roving, and top; yarn in single or plied, spun or filamentous form; threads, cords, and ropes but not metallic cables; yarn substitutes including twisted paper or foil, slit foil, or slit laminated sheets, metallic or plastic monofilaments.

(*3*) Products made from materials included in (*1*) or (*2*) or both by interlacing in any manner including weaving, knitting, tufting and braiding, bonding, knotting or felting and including such products as cloths, woven goods, knit goods, lace, embroidery, nets, webbing, batts, and nonwoven fabrics. Reinforced plastics and papers are not included.

(*4*) Wearing apparel, household and industrial articles fabricated principally from the material listed in (*1*), (*2*) and (*3*) above.

Appendix IV.—Replace Table I with a new table as shown in the accompanying Table IV.[5]

In Table II C, revise the portion of the table on "Cotton" to read as shown in the accompanying Table V.[5]

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Recommended Practice for:

Lighting Cotton Classing Rooms for Color Grading (D 1684 – 59 T).

Tentative Methods of Test for:

Heat Aging of Asbestos Textiles (D 1573 – 58 T), and

Fiber Length of Wool (D 1575 – 58 T).

ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that the Tentative Method of Test for Flammability of Clothing Textiles (**D 1230 – 52 T**) be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

Section 7.—In the second sentence change "4 sec" to "3½ sec."

Section 8(c).—In Class 1(*a*), change "4 sec" to "3½ sec."

In Class 2(*a*), change "4 to 7 sec" to "3½ to 7 sec."

In Class 3(*a*) and (*b*), change "4 sec" to "3½ sec."

REVISION OF STANDARDS IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard General Methods of Testing Woven Fabrics (D 39 – 59): Revise as appended hereto.[4]

Standard Specifications and Methods of Test for Fineness of Wool (D 419 – 58): Revise as appended hereto.[4]

Standard Methods of Testing Felt (D 461 – 59):

Section 18.—Delete this section and replace it with the following:

18. *Fiber Identification.*—(*a*) *Purpose.*—The purpose of this section is to provide a means for

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[5] Not included here; see revised Definitions, 1961 Book of Standards, Part 10.

analysis and identification of the fibers present in a felt beyond that information obtained in accordance with Section 21 (see Note 8 under Section 21).

(*b*) *Procedure.*—The felt sample shall be subjected to the appropriate analysis procedures set forth in the Methods for Quantitative Analysis of Textiles (ASTM Designation: D 629)[3] or Methods for Identification of Fibers in Textiles (ASTM Designation: D 276)[3], which procedures for any given felt sample may involve microscopic analysis, chemical analysis, or both.

Section 23.—Change the boldface heading to read "Acid Content in Felt (Pyridine Extraction Method)."

In Paragraph (*e*) transfer the reference to Note 9 from the first sentence to the end of the last sentence. Change Note 9 to read as follows:

NOTE 9.—A method alternative to the use of phenolphthalein is the titration to an end point of pH 8.3 potentiometrically using a calibrated glass electrode. This alternative method must be used where color extracted from the felt causes difficulty in determining a colorimetric end point.

Section 26.—In Paragraph (*c*), delete Items (6) and (7).

In the first sentence of Paragraph (*d*), change "The samples" to read "The sample."

Change Paragraphs (*e*) and (*g*) to read as follows:

(*e*) *Test Specimens.*—Six test specimens 2 in. in width by 12½ in. in length shall be cut and tested. Three of the test specimens shall be cut with the long dimension parallel to length of original roll, and three test specimens cut with long dimension parallel to width of original roll.

(*g*) *Procedure:*

(*1*) *Adjustment of Flame.*—Close off the air inlets at the base of the burner, and regulate the gas flow at the burner so that a luminous flame extending 1½ in. above the top of the burner is obtained.

(*2*) *Duration of Flaming.*—Suspend the specimen vertically ¾ in. above the top of the gas burner, with ½ in. of the upper end in the clamp, so that 12-in. length is exposed. Apply the flame vertically near the middle of the width of the lower end of the specimen for 12 sec, then withdraw it and record the time during which flaming continues on the specimen.

(*3*) *Time of Glow.*—The time of glow shall be the time the specimen continues to glow after it has ceased to flame. Record the time of glow in seconds.

Section 27.—Change to read as follows:

27. *Report.*—The average duration of flaming and time of glow shall be the average of the results obtained from the specimen tested in each of the length and width direction and shall be reported separately.

Standard Specifications and Method of Test for Fineness of Wool Tops (D 472 - 56): Revise as appended hereto.[4]

Standard Methods of Testing and Tolerances for Glass Yarns (D 578 - 58):[6] Revise as appended hereto.[4]

Standard Specifications and Method of Test for Fineness of Mohair Tops (D 1381 - 59): Revise as appended hereto.[4]

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

Revisions of the following standards as indicated have been published as tentative. They have been incorporated in the revised methods appended hereto:[4]

Standard General Methods of Testing Woven Fabrics (D 39 - 59), Sections 3 to 14 and 17 and 18.

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following standards and tentatives:

Tentative Specification for Fire-Retardant Properties of Treated Textile Fabrics (D 626 - 55 T):

Section 6.—Add the following new Paragraph (*a*) relettering present Paragraphs (*a*) and (*b*) as (*b*) and (*c*):

(*a*) *Adjustment of Flame.*—Close off the air inlets at the base of the burner, and regulate the gas flow at the burner so that a luminous flame extending 1½ in. above the top of the burner is obtained.

Standard Method of Test for Compatibility of Glass Yarn with Insulating Varnish (D 886 - 49):

Title and Scope.—Delete the word "Compatibility" and substitute the words "Impregnation and Penetration."

Standard Method of Core Sampling of Raw Wool in Packages for Determination of Percentage of Clean Wool Fiber Present (D 1060 - 58):

Section 2(a).—Add a new item (*1*) as follows, renumbering present items (*1*) and (*2*) as (*2*) and (*3*):

> (*1*) Each package shall have been uniformly and randomly packed, as evidenced by the absence of systematic differences in clean wool fiber present between different parts of the package.

In the present Item (*1*) substitute "(Note 6, Appendix)" for "(Note 4, Appendix)."

Section 3(a).—Change to read as follows:

> 3. (*a*) *Sampling Tool.*—The sampling tool shall consist of a tube equipped with a cutting edge, a drill, hammer, press or similar device, and accessories. It shall be capable of penetrating the required distance (Section 4(*b*)) into a package of wool and cutting a core therefrom, which core shall be retained substantially unchanged within the tube during its withdrawal from the package. Figure 1 illustrates the design of a recommended type of rotatable small-diameter wool-sampling tube for use in sampling baled wool of moderate density (Notes 1, 2, and 3).
>
> NOTE 1.—Sampling tubes in common use range from approximately ½ to 3 in. in diameter, and from 10 to 40 in. in length.
>
> NOTE 2.—A rotating tube is not recommended for use under circumstances where excessive heat will be generated.
>
> NOTE 3.—A toothed cutting edge on a rotating tube of small diameter, instead of the smooth edge shown in Fig. 1, is acceptable provided that for any specific design it has been shown that a bias is not introduced thereby. The absence of bias in samples obtained with a rotating 2-in. diameter tube with toothed cutting edge has been demonstrated.

Present Notes 2 and 3.—Renumber as Notes 4 and 5, respectively.

Section 5(e).—In the third sentence, insert "immediately" before "prior" and substitute "1961" for "19—."

Appendix A1.—Substitute "Note 6" for "Note 4" and "1961" for "19—;" insert "immediately" before "prior;" delete the second sentence beginning with the words "Any subsequent material . . ."

Table II.—Delete in their entirety the sections of the table headed "B. *Apparel Wool, Domestic*" and "C. *Carpet Wool.*"

Tentative Method of Test for Abrasion Resistance of Textile Yarns (D 1379 - 55 T):

Title.—Change to read "Tentative Recommended Practice for Operating Machine for Testing Abrasion Resistance of Textile Yarns."

Section 1.—In Paragraphs (*a*) and (*b*) in the first line, change the word "method" to read "recommended practice." In Paragraph (*e*) change the word "quantitatively" to read "qualitatively."

Section 3.—Change the boldface heading to read "Principle." In the first sentence change "abrasion method" to read "abrasion test."

REPORT OF COMMITTEE D-14

ON

ADHESIVES*

Committee D-14 on Adhesives held two meetings during the year: on Oct. 13 and 14, 1960, in Chicago, Ill., and on March 16 and 17, 1961, in Washington, D. C.

At the fall meeting, an informal talk was given by W. Thompson of the Argonne Laboratories on "Design of Remote Control Units for Reactors." At the spring meeting, R. Stromberg of the National Bureau of Standards spoke on "Adsorption of Polymers."

At the spring meeting, the committee voted to accept the responsibility of administering an ASTM Adhesives Award under the sponsorship of *Adhesives Age*. An Award Committee was appointed to determine procedure and to select a recipient for the first award.

The committee consists of 140 members, of whom 54 are classified as producers, 60 as consumers, and 26 as general interest members.

Due to the death of the Chairman, J. E. Rutzler, Jr., a new election for chairman and vice-chairman was held at the fall meeting which resulted as follows:

Chairman, A. A. Marra.

Vice-Chairman, N. J. DeLollis.

In line with a reorganization of Committee D-14 toward well-defined end use or materials subcommittees, supported by service subcommittees, Subcommittee V on Specifications which has been both, now has been reorganized to concern itself with end-use considerations of special adhesives that do not command the attention of the other materials subcommittees. Subcommittee V on Special Adhesives will function also as an initiator of new end-use groups. The section on Sampling has been transferred to Subcommittee I on Strength Tests.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1960 Annual Meeting, Committee D-14 presented to the Society through the Administrative Committee on Standards a proposed Tentative Recommended Practice for Atmospheric Exposure of Adhesive-Bonded Joints and Structures. The recommended practice was accepted by the Society on May 10, 1961, and has been assigned the designation D 1828 – 61 T.[1]

NEW TENTATIVES

The committee recommends for publication as tentative the following methods, specification, and recommended practice as appended hereto:[1]

Tentative Methods of Test for:

Permanence of Adhesive-Bonded Joints in Plywood Under Mold Conditions,

Peel Resistance of Adhesives (T-Peel Test),

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] The new tentatives appear in the 1961 Book of ASTM Standards, Part 6.

Density of Adhesives in Fluid Form, and Pressure-Sensitive Tack of Adhesives.

Tentative Specification for:

Water- or Solvent-Soluble Liquid Adhesives for Automatic Machine Sealing of Top Flaps of Fiberboard Shipping Cases.

Tentative Recommended Practice for:

Exposure of Adhesive Specimens to High-Energy Radiation.

REVISIONS OF TENTATIVE

The committee recommends revisions as follows of the Tentative Methods of Test for Resistance of Adhesives for Wood to Cyclic Laboratory Aging Conditions (**D 1183 – 55 T**)[2] and continuation of the methods as tentative.

Title.—Change to read: "Tentative Methods of Test for Resistance of Adhesives to Cyclic Laboratory Aging Conditions."

Section 1.—In the first sentence delete the words "for wood." Change the third sentence to read as follows:

> This method is intended for use with specimens described in approved strength test methods for adhesives such as the Method of Test for Cleavage Strength of Metal-to-Metal Adhesives (ASTM Designation: D 1062); Method of Test for Impact Strength of Adhesives (ASTM Designation: D 950); Method of Test for Peel or Stripping Strength of Adhesives (ASTM Designation: D 903); Method of Test for Strength of Adhesives on Flexural Loading (ASTM Designation: D 1184); Method of Test for Strength Properties of Adhesives in Plywood Type Construction in Shear by Tension Loading (ASTM Designation: D 906); Tentative Method of Test for Strength Properties of Adhesives in Shear by Tension Loading (Metal-to-Metal) (ASTM Designation: D 1002); Method of Test for Tensile Properties of Adhesives (ASTM Designation: D 897); and the Method of Testing Cross-Lap Specimens for Tensile Properties of Adhesives (ASTM Designation: D 1344).

TENTATIVE REVISIONS OF STANDARD

The committee recommends tentative revisions as follows of the Standard Definitions of Terms Relating to Adhesives (**D 907 – 60**):[2]

Add the following new definitions:

Failure, Adhesive.—Rupture of an adhesive bond, such that the plane of separation appears to be at the adhesive-adherend interface (See *Failure, Cohesive* and *Failure, Adherend.*)

Failure, Cohesive.—Rupture of an adhesive bond, such that the separation appears to be within the adhesive (See *Failure, Adhesive* and *Failure, Adherend.*)

Failure, Adherend.—Rupture of an adhesive bond, such that the separation appears to be within the adherend (See *Failure, Adhesive* and *Failure, Cohesive.*)

NOTE.—Examples of adherend failure are wood failure and fiber tear in paper.

Failure, Contact.—Failure of an adhesive joint due to incomplete contact, during bonding, between adhesive and adherend or between adhesive surfaces.

NOTE.—Incomplete contact may be due to insufficient flow or fusion of adhesive, nonuniform application of adhesive, poorly fitting adherends, etc.

Film, Adhesive, Supported.—An adhesive supplied in a sheet or in a film form with an incorporated carrier that remains in the bond when the adhesive is applied and used.

NOTE.—The carrier may be woven or nonwoven material comprised of organic or inorganic fibers.

Film, Adhesive, Unsupported.—An adhesive supplied in sheet or film form without an incorporated carrier.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Method of Test for:

Measuring the Hydrogen Ion Concentration of Dry Adhesive Film (**D 1583 – 58 T**),[2] and

Effect of Moisture and Temperature on Adhesive Bonds (**D 1151 – 58 T**).[2]

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

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[2] 1958 Book of ASTM Standards, Part 6.

Standard Methods of Test for:

- Tensile Properties of Adhesives (**D 897 – 49**),
- Applied Weight per Unit Area of Dried Adhesive Solids (**D 898 – 51**),
- Applied Weight per Unit Area of Liquid Adhesive (**D 899 – 51**),
- Peel or Stripping Strength of Adhesives (**D 903 – 49**),
- Impact Strength on Adhesives (**D 950 – 54**),
- Cleavage Strength of Metal-to-Metal Adhesives (**D 1062 – 51**),
- Blocking Point of Potentially Adhesive Layers (**D 1146 – 53**), and
- Effect of Bacterial Contamination, on Permanence of Adhesive Preparations and Adhesive Bonds (**D 1174 – 55**).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Methods of Test for:

- Strength Properties of Adhesives in Shear by Tension Loading (Metal-to-Metal) (**D 1002 – 53 T**),
- Susceptibility of Dry Adhesive Films to Attack by Roaches (**D 1382 – 55 T**),
- Susceptibility of Dry Adhesive Films to Attack by Laboratory Rats (**D 1383 – 55 T**), and
- Nonvolatile Content of Urea-Formaldehyde Resin Solutions (**D 1490 – 57 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Strength Tests (C. Dahlquist, chairman), has completed the proposed Tentative Method of Test for Peel Resistance of Adhesives (T-Peel Test) referred to earlier in this report. The subcommittee is continuing to work on improvement of the butt tension method, the torsional shear method, and the compression shear method. Work is also in progress on a static cleavage method.

Subcommittee III on Permanence (H. R. Butzlaff, chairman), has completed three recommendations: a revision of Methods D 1183, the proposed Tentative Method of Test for Permanence of Adhesive-Bonded Joints in Plywood Under Mold Conditions; and the new Tentative Recommended Practice for Atmospheric Exposure of Adhesive-Bonded Joints and Structures (D 1828 – 61 T), all referred to earlier in this report. Work is also in progress to revise Method D 896.

Subcommittee IV on Working Properties (N. J. DeLollis, chairman), has two methods, on determination of flow and on penetration, ready to submit to Committee D-14 letter ballot. Action is getting under way on a method to determine open assembly time.

Subcommittee V on Special Adhesives (M. Petronio, chairman), has completed a specification on poly (vinyl acetate) resin emulsion adhesive and it is ready for Committee D-14 letter ballot. Activity continues on developing specifications for general purpose (mending) adhesives, optical adhesives, and shoe adhesives.

Subcommittee VI on Nomenclature (F. H. Wetzel, chairman), continues to develop definitions of "maturing temperature," "fillet," "telegraphing," and "photographing" after two attempts to get approval of Committee D-14. The Subcommittee has written definitions for "batch," "manufactured unit," "solvent - activated adhesive," "heat-activated adhesive," "dry bond adhesive," and "multiple layer adhesive" for committee approval.

Subcommittee VII on Research Problems (C. B. Hemming, chairman), continues to explore the subject of adhesion —what is it? F. W. Reinhart led a very lively discussion at the October meeting on this subject.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee VIII on Electrical Properties (G. F. Lipsey, chairman), has prepared a tentative method for measuring conductivity of conductive adhesives for subcommittee letter ballot.

Subcommittee X on Wood Adhesives (R. F. Blomquist, chairman), is considering a revision of Method D 905. The effect of the angle of the grain of the wood to the glue line has been studied and suggests the need for some revision in cutting test specimens in this test. Also under consideration are modification of existing delamination tests to be applicable directly to evaluation of adhesives used. Revision of Method D 906 is under review. Particular items being considered are the change in thickness of veneer required and changes in cutting test specimens from bonded panels to compensate for the effects of lathe checks in the veneer on joint strength.

A method to determine the free formaldehyde content of particle board binders is under review. Consideration is being given to methods of evaluating binders without making and testing boards. Creep tests and peel tests are being reviewed and modified for use in evaluating wood adhesives, particularly for new bonded wood products.

The West Coast group of this subcommittee has revised Method D 1490 for committee approval.

Subcommittee XI on Metal Bonding Adhesives (R. L. Patrick, chairman).—The West Coast group under S. Yurenka has prepared four test methods and has submitted them to the East Coast group for review, editing, and submission to letter ballot of the sub committee: creep properties of adhesives in tensile shear in the temperature range of −67 F to 500 F (metal-to-metal); strength properties of adhesives in shear by compression loading (metal - to - metal); strength properties of adhesives in shear by tension loading in the temperature range of 600 F to 1500 F (metal-to-metal); and creep properties of adhesives in shear by compression loading (metal-to-metal).

Subcommittee XII on Adhesives for Plastics (M. Bodnar, acting chairman), is working with Subcommittee I to broaden certain test methods to make them apply also to the testing of bonded plastics. Methods being considered initially for revision include D 897 and D 1002; the suggested revisions will be submitted to subcommittee letter ballot prior to the next meeting.

A recommended practice for preparation of surfaces of plastics prior to adhesive bonding has passed D-14 letter ballot with minor revisions indicated.

This report has been submitted to letter ballot of the committee, which consists of 140 members; 99 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

A. A. MARRA,
Chairman.

G. S. CASEBOLT,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-14 presented to the Society through the Administrative Committee on Standards the recommendation that the Method of Test for Penetration of Adhesives be published as tentative. This recommendation was accepted by the Standards Committee on November 7, 1961, and the new method is available as a separate reprint bearing the designation D 1916 – 61 T.

REPORT OF COMMITTEE D-15
ON
ENGINE ANTIFREEZES*

Committee D-15 on Engine Antifreezes held one meeting during the year: April 12, 1961, in Washington, D. C. Subcommittee meetings were held on November 9 and 10, 1960, in New York City and on April 11 and 12, 1961, in Washington, D. C.

Six members resigned and eight members were elected to membership on the committee during the year. The committee now has 54 participating individuals, representing 39 voting members, of whom 13 are classified as producer, 17 are classified as consumer, and 9 as general interest members.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto:[1]

Tentative Methods of:

Glassware Test for Foaming Tendencies of Engine Antifreezes, and

Test for Effect of Antifreeze Solutions on Organic Finishes for Automotive Vehicles.

Tentative Recommended Practice for:

Selection of Engine Antifreeze for Use in Automotive Cooling Systems, Ethylene Glycol and Methanol Types.

REVISION OF TENTATIVE

The committee recommends revisions as appended hereto[2] of the Tentative Method for Glassware Corrosion Test for Engine Antifreezes (**D 1384 - 55 T**)[3] and continuance of the method as tentative pending consideration of further revisions.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Specifications for Hydrometer-Thermometer Field Tester for Engine Antifreezes (**D 1124 - 58 T**)[3] be approved for reference to letter ballot of the Society for adoption as standard without revision.

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for more than six years without revision.

Standard Method of Test for:

Freezing Point of Aqueous Engine Antifreeze Solution (**D 1177 - 54**),

Boiling Point of Engine Antifreezes (**D 1120 - 53**), and

Reserve Alkalinity of Concentrated Engine Antifreezes (**D 1121 - 54**).

Standard Method for:

Sampling and Preparing Aqueous Solutions of Engine Antifreeze for Testing Purposes (**D 1176 - 54**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

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* Sixty-fourth Annual Meeting of the Society, June 25-30, 1961.

[1] The new tentatives appear in the 1961 Book of ASTM Standards, Part 8.

[2] The revised method appears in the 1961 Book of ASTM Standards, Part 8.

[3] 1958 Book of ASTM Standards, Part 8.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee III on Physical Properties (C. M. White, chairman) prepared the Method for the Effect of Antifreeze on Organic Finishes for Automotive Vehicles referred to earlier in the report.

Subcommittee IV on Chemical Properties (E. F. Harford, Chairman):

Study Group on Glycol Analysis (J. W. Compton, chairman) has considered gas chromatographic methods for the analysis of glycols, and is preparing an article on this subject for publication in *Materials Research & Standards.*

Subcommittee V on Effect of Antifreeze on Rubber Hose (R. C. Barker, chairman) found that burst test methods under investigation appeared to evaluate the reinforcing fabric rather than the effect sought.

Subcommittee VI on Simulated and Actual Service Testing (C. H. Sweatt, chairman):

Study Group on Glassware Test Methods (E. L. Gibson, chairman) prepared the revision of the Glassware Corrosion Test (D 1384), referred to earlier in the report, in the light of new conditions, materials, and practices, and has further work in view. This group also prepared the new Glassware Foaming Test which is referred to earlier.

Study Group on Simulated Service Test Methods (V. O. Hatch, chairman) has conducted two interlaboratory test studies on the apparatus previously adopted and is making changes suggested by the revisions of the Glassware Corrosion Test (D 1384).

Subcommittee VII (B. E. Tiffany, chairman) prepared the Recommended Practice for the Selection of Antifreeze referred to earlier in the report. It has also been working with the Society of Automotive Engineers in the preparation of a report on specifying engine antifreezes.

Subcommittee VIII on Editorial and Coordination of Test Methods (Gus Kaufman, chairman) recommended the revision of *ASTM STP No. 120* referred to below.

Study Group on Revision of STP No. 120 on Selection and Use of Engine Antifreeze (C. M. White, Chairman) is revising and bringing up to date the information contained in Special Technical Publication No. 120 on The Selection and Use of Engine Antifreezes.

This report has been submitted to letter ballot of the committee, which consists of 39 voting members; 37 members returned their ballots, of whom 36 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

R. E. VOGEL,
Chairman.

W. D. MCMASTER,
Secretary.

REPORT OF COMMITTEE D-16
ON
INDUSTRIAL AROMATIC HYDROCARBONS AND RELATED MATERIALS*

Committee D-16 on Industrial Aromatic Hydrocarbons and Related Materials and its subcommittees held two meetings during the year: on June 29 to July 1, 1960, in Atlantic City, N. J., and on February 8 to 10, 1961, in Philadelphia, Pa.

At the close of the February meeting, the committee consisted of 89 members, of whom 68 were voting members; 34 were classified as producers, 27 as consumers, and 7 as general interest members.

PROPOSED METHODS TO BE PUBLISHED AS INFORMATION

The committee recommends for publication as information only the following proposed methods:[1]

Proposed Method of Test for:

Aldehydes in Styrene Monomer,
Solubility of Styrene Polymer,
Inhibitor, 4-*Tert*-Butylcatechol in Styrene Monomer, and
Polymer Content of Styrene Monomer.

REVISION OF TENTATIVE

The committee recommends revisions as follows of the Tentative Method of Test for Traces of Thiophene in Benzene Using Isatin and Spectrophotometry (**D 1685 – 59 T**):[2]

Section 6.—Change the heading of this section to read, "Preparation of Reagent Blank and Sample Blank." Change the first sentence of Section 6 (*b*) to read, "Prepare a sample blank in accordance with the procedure of Section 8 (*b*), except that the isatin shall not be added to the sample or to the reagent blank."

Section 8(e).—Change the first sentence to read, "Determine the absorbance of the sample blank using the reagent blank without isatin present as a reference (Section 6(*b*))."

Section 9.—Add a new Table I as shown in the accompanying Table I. Add a reference to this new Table I at the end of the definitions for A and B in the equation. Renumber the present Table I as Table II.

Section 10.—Change Note 2 to read as follows:

NOTE 2.—The precision limits given in Table II are based on data published in the Appendix to D 1685, ASTM Standards, Part 8, 1959 Supplement.

Appendix.—Delete the appendix.

Capitalize or use italics for the words "Sample Blank" and "Reagent Blank" wherever they appear throughout the text.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Method of Test for Color of Solid Aromatic Hydrocarbons and Re-

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] The proposed methods appear in the 1961 Compilation of ASTM Standards on Benzene, Toluene, Xylene, and Solvent Naphtha.

[2] 1959 Supplement to Book of ASTM Standards, Part 8.

TABLE I.

Quantity in Equation of Section 9	Spectrophotometric Cell Solutions		
	Solution in Cell	versus	Solution in Reference Cell
A =	H_2SO_4; $Fe_2(SO_4)_3$; Isatin (after contact with sample, Sect. 8(*b*))	versus	H_2SO_4; $Fe_2(SO_4)_3$; Isatin (Reagent Blank, Sect. 6(*a*))
B =	H_2SO_4; $Fe_2(SO_4)_3$ (after contact with sample—sample blank, Sect. 6(*b*))	versus	H_2SO_4; $Fe_2(SO_4)_3$ (Reagent Blank without isatin, Sect. 8(*e*))

lated Materials in the Molten State (Platinum-Cobalt Scale) **(D 1686 - 59 T)**[2] be approved for reference to letter ballot of the Society for adoption as standard without revision.

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following tentative and standard:

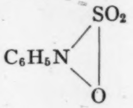
Standard Method of Test for Distillation of Industrial Aromatic Hydrocarbons (D 850 - 56):[3]

Table I.—Extend the table as follows:

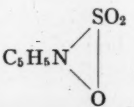
ASTM Thermometer	Name	Range, deg Cent	Subdivision, deg Cent
102C	Solvents distillation	123 to 177	0.2
103C	Solvents distillation	148 to 202	0.2
104C	Solvents distillation	173 to 227	0.2
105C	Solvents distillation	198 to 252	0.2

Tentative Method of Test for Water in Phenol and Related Materials by the Iodine Reagent Method (D 1631 - 59 T):[2]

Section 2.—Change



to read



Note 1.—Change to read as follows:

NOTE 1.—In place of the divided reagents described in Sections 5(*a*) and (*c*) it is permissible to employ the single-solution reagent specified in the Method of Test for Water in Lacquer Solvents and Diluents (ASTM Designation: D 1364). This reagent should be used with a sample solvent consisting of pyridine with or without methanol or ethylene glycol. If ketones are present in the sample, interference from them can be avoided by employing the D 1364 reagent together with the glycol-pyridine sample solvent specified in that method.

Note 2.—Change to read as follows:

NOTE 2.—Stabilized divided and single-solution Karl Fischer reagents are available from laboratory supply houses, and may also be used.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee A on Monocyclic Aromatics (K. M. Brown, chairman) has conducted collaborative testing on a revised shaking procedure for Method D 848 and on a colorimetric method for the determination of trace amounts of carbon disulfide in benzene. The subcommittee is also considering revisions of Methods D 1685, D 891, and D 1555 as well as expansion of scope of the latter method to include styrene. The styrene task group plans collaborative testing of the methods for inhibitor, aldehydes, polymer content, and solubility of polymer and is preparing methods for chlorine, sulfur, and peroxides in styrene.

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[3] 1958 Book of ASTM Standards, Part 8.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Task groups are investigating gas chromatographic procedures for saturated impurities and methods for trace sulfur in benzene, toluene, and xylene. New task groups have been formed to study the suitability of present ASTM specifications for benzene, toluene, and xylene and to devise a gas chromatographic method for xylene isomers.

Subcommittee B on Polycyclic Aromatics (M. Mitchell, chairman) has conducted further cooperative tests of refined naphthalene solidification points using the National Bureau of Standards naphthalene thermometric standard. A proposed method for evaporation residue of crude naphthalene will soon undergo collaborative testing. A proposed photometric method for acid wash color of naphthalene is in preparation. The determination of sulfur in crude naphthalene is also being investigated.

Subcommittee C on Phenolic Compounds (D. F. Pontz, chairman) has cooperatively tested a cloud point method for water solubility of phenol. Also undergoing study are methods for water content, distillation, oil content, and solidification point of tar acids. A task group continues activity on phenol assay.

Subcommittee D on Nitrogen Heterocyclics (R. F. Shertzer, chairman) is continuing the development of methods for color, water solubility, reducing substances, and oil content of refined pyridine.

Subcommittee E on Thermometry (M. Lapeyrouse, chairman) has continued activity on problems related to the National Bureau of Standards' thermometric standards, particularly the one for phthalic anhydride.

Progress has also been made in selecting a series of overlapping narrow range partial immersion distillation thermometers to replace thermometer 42C and in selecting a wide-range thermometer for tar acids distillations.

Subcommittee F on Editorial and Nomenclature (E. T. Scafe, chairman) editorially reviewed all methods devised by the other subcommittees and prepared new drafts of scope and the regulations governing Committee D-16.

Subcommittee G on Statistical Procedure (W. J. Tancig, chairman) served as a consultant to other subcommittees with statistical problems.

Subcommittee H, American Group for ISO/TC 78 on Aromatic Hydrocarbons (K. M. Brown, chairman) has prepared comments on draft British standard specifications and prepared a draft of a proposed procedure for handling ISO TC/78 matters with the ASA and ASTM Committee D-16.

Subcommittee I on Phthalic Anhydride (O. E. Knapp, chairman) has adopted an aluminum heating block and will soon begin collaborative testing of the color of phthalic anhydride in the molten state and after heating. Phthalic anhydride solidification point testing awaits resolution of problems with the thermometric standard.

Subcommittee J on Handling and Sampling (W. J. Tancig, chairman) was organized in February, 1961, and is now formulating a program of work.

This report has been submitted to letter ballot of the committee, which consists of 66 voting members; 57 members returned their ballots, of whom 48 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. E. SISCO,
Chairman.

K. H. FERBER,
Secretary.

REPORT OF COMMITTEE D-17
ON
NAVAL STORES*

Committee D-17 on Naval Stores held two meetings during the year: on June 29, 1960, in Atlantic City, N. J., and on February 28, 1961, in Jacksonville, Fla. The latter meeting was held jointly with the Technical Committee, Tall Oil Division of the Pulp Chemicals Assn. in view of the mutual interest of the two groups in tall oil and rosin products.

The following new members were added to the committee during the year: B. L. Hampton, Nelio Resins Inc.; H. E. McLaughlin, Newport Industries; E. A. Bried, Hercules Powder Co.; and R. A. Reck, Armour Industrial Chemical Co. (replacing V. A. Franklin, deceased). Resignations were received from H. J. Blank, Zophar Mills; N. R. Craig, U. S. Dept. of Agriculture; L. A. Goldblatt, U. S. Dept. of Agriculture; Foster Dee Snell, Foster Dee Snell, Inc.; and I. E. Knapp, retired.

The committee consists of 27 members, of whom 11 are classified as producers, 7 as consumers, and 9 as general interest members.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot vote of the Society:

Standard Methods of Testing Tall Oil (D 803 - 58):[1]

Section 6.—Replace this section with the following new Sections 6, 7, and 8, renumbering the present Sections 7 and 8 and subsequent sections accordingly:

Apparatus

6. The viscosity of tall oils that contain no crystals at 25 C can be measured by means of the Gardner-Holdt bubble tubes. These tubes shall be of clear glass with a closed, flat, even bottom and shall have the following approximate dimensions and markings:

(*1*) A uniform internal diameter throughout the length of the tube of 10.65 ml,

(*2*) An internal length of 112 mm,

(*3*) An etched line around the outside of the tube 5 mm from the open end, and

(*4*) A second etched line around the outside of the tube 13 mm from the open end.

The tubes shall provide results that are in agreement with the data shown in Table I.

Procedure

7. (*a*) Fill the tube with the sample, free of solid particles or air bubbles, so that the apparent upper edge of the liquid meniscus is even with the lower etched line on the tube.

(*b*) Bring the filled, stoppered tube to equilibrium at 25 ± 0.25 C (77 ± 0.45 F). Invert the tube and allow to remain inverted for twice the anticipated bubble time in order to bring the bubble to complete rest in the inverted tube. Quickly revert the tube to upright position, still at 25 C and, with the tube exactly vertical, carefully note the time, to 0.1 sec, for the bubble to rise until the first sign of its deformation against the cork. This procedure is repeated and the two times averaged.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] 1958 Book of ASTM Standards, Part 8.

Report

8. (*a*) The average of the two times is reported as the viscosity in Gardner seconds. The result is reported to the nearest 0.1 sec if the time is less than 10.0 sec, and to the nearest second if the time is 10.0 sec or more. This time, in seconds, may be translated to viscosity in stokes as follows:

$$V_s = 0.73T - \frac{0.5}{T^2}$$

where:

V_s = viscosity in stokes, and

T = time in seconds.

Reasonable accuracy is obtained by multiplying time in seconds by 0.73 on materials having a viscosity greater than 1.5 stokes.

(*b*) *Precision.*—When using this method an operator can duplicate results to within ±5 per cent, and agreement between different operators is within ±10 per cent, except at the limits of this method of measurement.

(*c*) For convenience, the results are sometimes reported by Gardner-Holdt letters. These are as defined in Table I.

TABLE I.—GARDNER-HOLDT LETTER DEFINITIONS OF VISCOSITY REFERENCE STANDARDS.

(New Table I, Methods D 803)

Letter	Stokes	Seconds	Letter	Stokes	Seconds
A5	0.00505	...	Q	4.35	6.0
A4	0.0624	...	R	4.70	6.4
A3	0.144	...	S	5.0	6.8
A2	0.220	...	T	5.5	7.6
A1	0.321	...	U	6.27	8.6
A	0.50	1.2	V	8.84	12.1
B	0.65	1.3	W	10.7	14.7
C	0.85	1.5	X	12.9	17.7
D	1.00	1.6	Y	17.6	24.0
E	1.25	1.9	Z	22.7	31.0
F	1.40	2.1	Z1	27.0	37.0
G	1.65	2.3	Z2	36.2	50.0
H	2.00	2.8	Z3	46.3	63.0
I	2.25	3.1	Z4	63.4	87.0
J	2.50	3.4	Z5	98.5	135.0
K	2.75	3.8	Z6	148	202.0
L	3.00	4.1	Z7	388	...
M	3.20	4.4	Z8	590	...
N	3.40	4.7	Z9	855	...
O	3.70	5.1	Z10	1066	...
P	4.00	5.5			

New Table I.—Add a new Table I as shown in the accompanying Table I.

Sections 9 to 12.—Delete these sections, which cover color by the Lovibond Method, deleting also Table I and Footnote 6.

Section 13.—Replace this section with the following new Sections 11, 12, and 13, renumbering the subsequent sections accordingly:

Color Reference Standards

11. (*a*) Primary standards for color comparison consist of 18 solutions of varying amounts of pure powdered $K_2Cr_2O_7$ dissolved in pure H_2SO_4 (sp gr 1.84). Each solution is prepared from the amounts prescribed in Table II. Gentle heat may be applied, if necessary, to effect solution of the dichromate.

(*b*) Since the $K_2Cr_2O_7$–H_2SO_4 solutions must be freshly made for this color comparison, it is more convenient to compare samples with a series of permanent solutions, which are called "Gardner-1933 Standards" and are defined as solutions blended in the proportions prescribed in Table II from the following stock solutions:

(*1*) *Ferric Chloride Solution.*—Prepare a solution containing approximately 5 parts by weight of $FeCl_3 \cdot 6H_2O$ and 1.2 parts of HCl (1:17). Adjust to exact color equivalence to a freshly prepared solution containing 3 g of $K_2Cr_2O_7$ in 100 ml of H_2SO_4 (sp gr 1.84).

(*2*) *Cobalt Chloride Solution.*—Prepare a solution containing 1 part by weight of $CoCl_2 \cdot 6H_2O$ to 3 parts of HCl (1:17).

(*3*) *Hydrochloric Acid* (*1:17*).

Procedure

12. Determine the color by comparison with the reference standard solutions prescribed in Table II, conveniently by comparing the sample and the standard in Gardner-Holdt viscosity tubes, described in Section 6. Make the comparison at 25 ± 5 C by placing the tubes close together and looking through them against a background which is substantially equal in color to the northern sky.

NOTE.—Secondary reference standards may be obtained in the form of 18 colored glass disks, which are set into a pair of larger, plastic disks and the latter mounted to rotate in a housing for holding the sample tube and glass disk in close and fixed proximity.[2]

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13. The color shall be reported in terms of one of the 18 Gardner standards that is nearest to it in color. For example, No. 4 would indicate that the color of the sample was nearest to that of Gardner standard No. 4.

New Table II.—Add a new Table II as shown in the accompanying Table II.

TABLE II.—REFERENCE STANDARD COLOR SOLUTIONS.
(New Table II, Methods D 803)

Gardner (1933) Color Standard No.	Composition of Gardner Standards			Composition of Primary Standards
	Ferric Chloride Solution, ml	Cobalt Chloride Solution, ml	Hydrogen Chloride Solution, ml	Potassium Dichromate, g per 100 ml of sulfuric acid
1	0.13	0.19	99.68	0.0039
2	0.19	0.29	99.52	0.0048
3	0.29	0.43	99.28	0.0071
4	0.43	0.65	98.92	0.0112
5	0.65	0.97	98.38	0.0205
6	1.00	1.3	97.7	0.0322
7	1.7	1.7	96.6	0.0384
8	2.5	2.0	95.5	0.0515
9	3.3	2.6	94.1	0.0780
10	5.1	3.6	91.3	0.164
11	7.5	5.3	87.2	0.250
12	10.3	7.6	81.6	0.380
13	16.6	10.0	73.4	0.572
14	22.2	13.3	64.5	0.763
15	29.4	17.6	53.0	1.041
16	37.8	22.8	39.4	1.280
17	51.3	25.6	23.1	2.220
18	100.0	0.0	0.0	3.000

Sections 51 to 55.—Delete these sections, which cover determination of rosin acids by the McNicoll Method, renumbering Sections 56 through 62 as Sections 51 through 57.

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Specifications for:

Spirits of Turpentine (**D 13 - 51**).

Method of:

Test for Toluene Insoluble Solid Matter in Rosin (Chiefly Sand, Chips, Dirt, and Bark) (**D 269 - 52**),
Sampling and Testing Pine Tars and Pine Tar Oils (**D 856 - 49**),
Test for Ash in Rosin (**D 1063 - 51**), and
Test for Rosin Acids in Fatty Acids (**D 1240 - 54**).

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Methods of Test for Fatty Acids Content of Tall Oil Rosin (**D 1585 - 58 T**).

EDITORIAL CHANGES

The committee recommends the following editorial change in the Standard Methods of Sampling and Grading Rosin (**D 509 - 55**):[1]

Section 2(e).—Add to Footnote 3 the following paragraphs:

> In circumstances where an applicant is unable to obtain the loan of a set of the U.S. Rosin Standards, because of the limited number of sets in existence, secondary sets are available. Lovibond "U.S." rosin standards are manufactured by Tintometer, Ltd., Salisbury, England. The United States agents for Tintometer, Ltd. is Hayes G. Shimp, Inc., 866 Willis Ave., Albertson, L. I., New York.
>
> These standards are excellent duplicates of the U.S. Rosin Standards but could not be classified as identical. The Lovibond standards do not have official recognition by the U.S. Department of Agriculture. A certificate may be obtained, however, at a cost of $10.00 from the Naval Stores Branch, Agricultural Marketing Service, attesting to the degree with which a set compares visually with the master set of the Official U.S. Rosin Color Standards."

The recommendations in this report

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[2] The pair of disks and a comparator housing may be obtained as Set 605-VC from Hellige, Inc., 3718 Northern Boulevard, Long Island City, N. Y. Although these disks are marked "Hellige" they should not be confused with the Hellige (1930) series of color standards which are designated 1L, 1, 2L, 2, etc.

have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Methods for most of the products under jurisdiction of the committee are fairly well established. Current activities of the subcommittees have been concerned mainly with tall oil and its products, on which the committee is working cooperatively with the Tall Oil Division of the Pulp Chemicals Assn.

Subcommittee IV on Chemical Analysis of Rosin (H. E. McLaughlin, chairman) has been studying the precision of results obtained in collaborative tests with Method D 1585 - 58 T, and is recommending continuation of this method as tentative. It is further recommending that Method D 509 - 55 be continued as standard with inclusion of a note on the availability of secondary rosin color standards; also that Methods D 269 - 52 and D 1163 - 51 be reapproved as standard without change.

Subcommittee V on Tall Oil (J. J. Owen, chairman) is recommending that Method D 803 - 56 be continued as standard with inclusion of the Gardner-Holdt Viscosity and the Gardner Color Methods previously referred to in Committee D-1's former Method D 154, and that the Lovibond Color and the McNicoll Rosin Acids Methods be deleted from Method D 803.

It is recommending that Method D 1240 - 54 be reapproved as standard without change.

The committee is cooperating with Group 7, Subcommittee IX of Committee D-1 on Paint, Varnish, Lacquer, and Related Products; and with the Technical Committee, Tall Oil Division of the Pulp Chemicals Assn. on developing a method or methods for determining rosin in coating vehicles.

Subcommittee VII on Terpene Hydrocarbons and Pine Oil (R. E. Price, chairman) is recommending that Specifications D 13 51 be reapproved as standard without change.

Subcommittee IX on Nomenclature and Definitions (J. L. Boyer, chairman) is cooperating with the Technical Committee, Tall Oil Division of the Pulp Chemicals Assn., in studying a glossary of terms and definitions for tall oil products for possible inclusion in Definitions D 804 - 57.

This report has been submitted to letter ballot of the committee, which consists of 26 members; 17 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

S. R. SNIDER,
Chairman.

W. A. KIRKLIN,
Secretary.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE D-18

ON

SOILS FOR ENGINEERING PURPOSES*

Committee D-18 on Soils for Engineering Purposes held one meeting in Atlantic City, N. J., on June 29, 1960, during the Annual Meeting of the Society. Executive Committee meetings were held in Atlantic City on June 27, 1960, and in Washington, D. C., on January 8, 1961.

During the Annual Meeting of the Society in Atlantic City, the following papers sessions were held: (1) General Soils Session, and (2) Symposium on Nuclear Methods for Measuring Soil Density and Moisture. The symposium has been published by the Society as a special technical publication, *ASTM STP No. 293*, and the papers from the general session have appeared in the 1960 *Proceedings*.

The following members of Committee D-18 passed away during the year and will be missed greatly by their colleagues: E. J. Felt, and H. F. Clemmer. Resignation was accepted during the year from J. A. Leadabrand, Portland Cement Assn. A. W. Maner was appointed to replace H. F. Clemmer on the Standing Committee on Simplified Practice Recommendation for Coarse Aggregates.

The committee consists of 148 members, of whom 116 are voting members; 23 are classified as producers; 40 as consumers; and 53 as general interest members.

Special Committee on Award of Merit.—The following persons were appointed to the special committee for award of merit for the coming year: D. M. Burmister, chairman; M. D. Catton, continuing member; and H. F. Clemmer, new member. On January 8, 1961, Mr. K. B. Woods was appointed to replace H. F. Clemmer, deceased October 5, 1960.

Special Committee for Hogentogler Award.—This special committee, consisting of D. M. Burmister (chairman), W. E. Schmid, and R. L. Schiffman, recommended that the Hogentogler Award be presented to C. B. Crawford for his paper entitled "The Influence of Rate of Strain on Effective Stresses in Sensitive Clay."

Symposia Committee—R. K. Bernhard, Chairman of Subcommittee R-9, and members of his subcommittee were appointed to arrange for a one-half day Symposium on Dynamic Properties of Soils to be held at the Annual Meeting in June 1961. This symposium is to be of the panel-discussion type.

Subcommittee G-4 on Environmental Conditions.—This administrative subcommittee was established on January 8, 1961, with D. M. Burmister as chairman. A name, scope, and proposed membership is to be submitted to the Executive Committee during the next regular meeting in June 1961.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1960 Annual Meeting, Committee D-18 presented to the

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

Society through the Administrative Committee on Standards the recommendation that the Definitions of Terms Relating to Soil Dynamics be published as tentative. This recommendation was accepted by the Standards Committee on September 23, 1960, and the definitions appear in the 1960 Supplement to Book of ASTM Standards, Part 4, bearing the designation D 1707 - 60 T.

NEW TENTATIVE

The committee recommends for publication as tentative the Method of Test for Bearing Ratio of Laboratory-Compacted Soils, as appended hereto.[1]

REVISION OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Method for Grain-Size Analysis of Soils (D 422 - 60 T):[2]

Section 1.—Add the following new Note 2 after this section, numbering the present note as "Note 1":

NOTE 2.—Two types of dispersion devices are provided: 1) a high-speed mechanical stirrer, and (2) air dispersion. Extensive investigations indicate that air dispersion devices produce a more positive dispersion of plastic soils below the 20-micron size and appreciably less degradation on all sizes when used with sandy soils. Because of the definite advantages favoring air dispersion, its use is recommended. The results from the two types of devices differ in magnitude, depending upon soil type, leading to marked differences in particle size distribution, especially for sizes finer than 20 microns.

Sections 7 and 10.—Replace "110 C (230 F)" with "110 ± 5 C (230 ± 9 F)."

Section 17(a).—Add the following new Item 6: "(6) The dispersion device used and length of the dispersion period."

Tentative Method of Test for Liquid Limit of Soils (D 423 - 59T):[3]

Section 1.—Renumber as Section 2, and change the heading from "Scope" to read "Definition."

New Section 1.—Add the following new Section 1 under the heading "Scope":

1. (*a*) This method of test provides for the determination of the liquid limit of soils as defined in Section 2, using the mechanical device specified in Section 3, securing the results of at least three trials, and the plotting of a flow curve. Provision is also made for: (*1*) a one-point method requiring the calculation of the liquid limit value from data secured from a single trial; and (*2*) a procedure for check or referee tests.

(*b*) The mechanical method shall be used unless specifications for the soil being tested permit the use of the one-point method. The procedure for check or referee tests shall be used in cases of disagreement or when required in the specifications for the soil being tested.

Sections 2 through 8.—Renumber the present Sections 2 through 8 as Sections 3 through 9. In the present Section 5(*d*), replace "110 C (230 F)" with "230 ± 9 F (110 ± 5 C)."

New Sections.—Add the following new Sections 10 and 11 under the centered heading "One-Point Method," renumbering subsequent sections accordingly:

Procedure

10. (*a*) The requirements for apparatus, sample, and adjustment of the mechanical device are the same as specified in Sections 3, 4. and 5.

(*b*) Follow the test procedure given in Section 6, (*a*) through (*e*), except that a moisture-content sample shall be taken only for the accepted trial. The accepted trial shall require between 20 and 30 drops of the cup to close the groove and at least two consistent consecutive closures shall be observed before taking the moisture-content sample for calculation of the liquid limit. The test shall always proceed from the dryer to the wetter condition of the soil.

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[1] The new method appears in the 1961 Book of ASTM Standards, Part 4.

[2] 1960 Supplement to Book of ASTM Standards, Part 4.

[3] 1958 Book of ASTM Standards, Part 4.

Calculations

11. (*a*) Calculate the water content, w_N, for the accepted trial, expressed as a percentage of the oven-dried weight, as follows:

$$w_N = \frac{\text{weight of water}}{\text{weight of oven-dried soil}} \times 100$$

(*b*) Determine the liquid limit, LL, using the following formula:

$$\text{LL} = w_N(N/25)^{0.12}$$

where:

N = number of drops of the cup required to close the groove at the water content, w_N.

Report the liquid limit value to the nearest whole number. Values of $(N/25)^{0.12}$ are given in Table I.

TABLE I.—VALUES OF $(N/25)^{0.12}$

N	$(N/25)^{0.12}$	N	$(N/25)^{0.12}$	N	$(N/25)^{0.12}$
20.....	0.974	24...	0.995	28...	1.014
21.....	0.979	25...	1.000	29...	1.018
22.....	0.985	26...	1.005	30...	1.022
23.....	0.990	27...	1.009		

New Section 14.—Add the following new Section 14 under the heading "Report":

> 14. The report shall include the following:
>
> (*1*) Liquid limit value,
>
> (2) Procedure followed when either the one-point method or the procedure for check or referee tests is used.

Tentative Method of Test for Load-Settlement Relationship for Individual Piles Under Vertical Axial Load (D 1143 – 57 T):[3]

Section 4.—Add the following at the end of Paragraph (*b*):

> Some suitable device shall be used to maintain constant tension in the wire throughout the test. This method of measuring settlement shall be used only as a check test on pile loading tests of minor importance.

Change the last sentence of Paragraph (*c*) to read as follows:

> While a dial gage may provide the greatest accuracy in consecutive settlement readings, a check settlement observation shall be made before and after the loading test and whenever possible at selected intervals during the test by level rod or scale referred to a fixed elevation which is independent of the reference beam to which the dial gage is attached.

Section 5(*b*).—In lines 7 and 12 change "0.001 ft" to read "0.01 in." The use of 0.001 ft in Section 4(*a*) will be retained, as level rods are so graduated and read in making level observations.

To provide for a fixed schedule of taking settlement readings between load increments, change the second sentence to read as follows:

> Settlement readings made to an accuracy of 0.01 in. shall be taken before and after the application of each new load increment and at 2, 4, 8, 15, 30, 60 min, and every 2 hr until application of the next load increment.

Change the fourth sentence to read as follows:

> When the loading has been completed the full test load shall remain on the pile for 24 hr or for a longer period if the necessity therefor is indicated by the rate of settlement of the pile, and settlement readings shall be taken at 6 hr intervals during the period and at the end of that period.

Add the following at the end of Paragraph (*b*), preceding Note 2:

> In testing friction piles in cohesive soil, a time period of not less than 24 hr shall elapse between driving and loading of the test pile; and a longer period, not to exceed two weeks, may be required if experimental data or other test conditions indicate this to be necessary. Constant attention of the operator or automatic control is required to maintain the specified jacking pressure at all times during the loading test.

ADOPTION OF TENTATIVE AS STANDARD WITH REVISIONS

The committee recommends that the Tentative Method of Testing Soil-Bituminous Mixtures (**D 915 – 47 T**)[3] be approved for reference to letter ballot of the Society for adoption as standard with editorial changes as indicated:

Section 1.—Change to read as follows:

1. *Scope.*—This method of test provides for the determination of water absorption, expansion, and extrusion characteristics of compacted soil or soil-aggregate mixtures containing liquid bituminous material. The method is intended for determining the effects of water on these mixtures. It may also be employed to determine the same characteristics for untreated soil or soil-aggregate mixtures. The results are not intended for use in formulas to determine pavement thickness, nor to predict relative field performance of different bituminous materials as stabilization admixtures.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as follows of the Standard Method of Test for Shrinkage Factors of Soils (**D 427 – 39**),[3] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

Section 1. — Change "Volumetric change" to "Volumetric shrinkage," and "Lineal shrinkage" to "Linear shrinkage."

Section 2.—In Paragraph (*a*) change "4½ in." to "5½ in." In Paragraph (*d*) change "12 in." to "6 in." In Paragraph (*e*) change "2 in." to "2¼ in." and "1 in." to "1¼ in." Replace the word "level" with: "is in a plane essentially parallel with the bottom of the cup."

Section 4(*c*).—Delete the word "level" in the second line. Change "110 C" to "230 ± 9 F."

Section 5.—Change the letter "*M*" to "*w*" in the formula.

Section 6.—Change Paragraph (*a*) to read: "The shrinkage limit of a soil is the maximum water content at which a reduction in water content will not cause a decrease in the volume of the soil mass." In Paragraph (*b*), change the letter "*S*" to the letters "*SL*" and the letter "*M*" to "*w*." In Paragraph (*c*), delete the word "true" in second line. Change "*G*" to "G_s." Change "*S*" to "*SL*."

Section 7(*a*).—Change to read as follows:

The shrinkage ratio of a soil is the ratio of: (1) a given volume change, expressed as a percentage of the dry volume, to (2) the corresponding change in water content above the shrinkage limit, expressed as a percentage of the weight of oven-dried soil.

Section 8.—Change the heading to read "Volumetric Shrinkage," and revise the Section to read as follows:

8. (*a*) The volumetric shrinkage of a soil is the decrease in volume, expressed as a percentage of the soil mass when dried, of a soil mass when the water content is reduced from a given percentage to the shrinkage limit.

(*b*) The volumetric shrinkage, V_s, shall be calculated from the data obtained in the volumetric shrinkage determination by the following formula:

$$V_s = (w_1 - SL)R$$

where:

w_1 = given percentage of water content,

SL = shrinkage limit, and

R = shrinkage ratio.

Section 9.—Change the heading to read "Linear Shrinkage," and revise the Section to read as follows:

9. (*a*) The linear shrinkage of a soil is the decrease in one dimension of a soil mass, expressed as a percentage of the original dimension, when the water content is reduced from a given value to the shrinkage limit.

(*b*) The linear shrinkage, L_s, shall be obtained by means of the following formula:

$$L_s = 100\left(1 - \sqrt[3]{\frac{100}{V_s + 100}}\right)$$

Figure 2.—Delete Fig. 2.

Section 10.—Change to read as follows:

10. The specific gravity of the soil solids, G_s, may be calculated from the data obtained in the volumetric shrinkage test by the following formula:

$$G_s = \frac{1}{1/R - SL/100}$$

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Specifications for:

Materials for Soil-Aggregate Subbase, Base, and Surface Courses (**D 1241 - 55 T**).

Tentative Methods of:

Soil Investigation and Sampling by Auger Borings (**D 1452 - 56 T**),
Penetration Test and Split-Barrel Sampling of Soils (**D 1586 - 58 T**),
Thin-Walled Tube Sampling of Soils (**D 1587 - 58 T**),
Test for Shear Strength of Flexible Road Surfaces, Subgrades, and Fills by the Burggraf Shear Apparatus (**D 916 - 47 T**),
Testing Soil-Bituminous Mixtures (**D 915 - 47 T**),
Test for Water-Soluble Chlorides Present as Admixes in Graded Aggregate Road Mixes (**D 1411 - 56 T**),
Test for Density of Soil in Place by the Sand-Cone Method (**D 1556 - 58 T**),
Test for Making and Curing Soil-Cement Compression and Flexure Test Specimens in the Laboratory (**D 1632 - 59 T**),
Test for Compressive Strength of Molded Soil-Cement Cylinders (**D 1633 - 59 T**),
Test for Compressive Strength of Soil-Cement Using Portions of Beams Broken in Flexure (Modified Cube Method) (**D 1634 - 59 T**), and
Test for Flexural Strength of Soil-Cement Using Simple Beam with Third-Point Loading (**D 1635 - 59 T**).

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following standards:

Standard Method of Test for Plastic Limit and Plasticity Index of Soils (D 424 - 59):[3]

Section 4(c).—Change "110 C (230 F)" to "230 ± 9 F (110 ± 5 C)."

Standard Method of Test for Centrifuge Moisture Equivalent of Soils (D 425 - 39):[3]

Section 5(b).—Change "110 C (230 F)" to "230 ± 9 F (110 ± 5 C)."

Standard Method of Test for Shrinkage Factors of Soils (D 427 - 39):[3]

Section 4(c).—Replace "110 C (230 F)" with "230 ± 9 F (110 ± 5 C)."

Standard Method of Test for Specific Gravity of Soils (D 854 - 58):[3]

Sections 5(b) and (c).—Replace "110 C" with "110 ± 5 C (230 ± 9 F)."

Standard Method of Test for Amount of Material in Soils Finer than the No. 200 Sieve (D 1140 - 54):[3]

Sections 4(a) and (e).—Replace "110 C (230 F)" with "230 ± 9 F (110 ± 5 C)."

Tentative Method of Test for Moisture-Penetration Resistance Relations of Fine-Grained Soils (D 1558 - 58 T):[3]

Section 6.—Change to read as follows:

6. Plot the penetration resistance values and the corresponding moisture contents (as calculated in accordance with Section 5) on the same graph sheet with the corresponding moisture-density relations data (as provided in Paragraph 12(*a*) of Method D 698). Plot the moisture-penetration resistance data immediately above the moisture-density data, using the same moisture content scale for both sets of data. The moisture-penetration resistance relationship curve shall be established by not less than three determinations.

Tentative Methods of Test for Moisture-Density Relations of Soils, Using a 5.5-lb Rammer and 12-in. Drop (D 698 - 58 T):[3]

Section 12.—Change Paragraphs (*a*) and (*b*) to read as follows:

12. (*a*) Plot the oven-dry weights per cubic foot (densities) of the soil as ordinates and the corresponding moisture contents as abscissas.

Draw a smooth curve connecting the plotted points.

(*b*) *Optimum Moisture Content*, w_o.—The moisture content corresponding to the peak of the curve drawn as provided in Paragraph (*a*) shall be termed the "optimum moisture content" of the soil under the above compaction.

Tentative Methods of Test for Moisture-Density Relations of Soils, Using a 10-lb Rammer and an 18-in. Drop (D 1557 – 58 T):[3]

Section 12.—Revise Paragraphs (*a*) and (*b*) as indicated above for Section 12 of Method D 698.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee G-1, Editorial (E. E. Bauer, chairman) has given editorial review to the proposed Tentative Method of Test for Determining the Bearing Ratio of Laboratory-Compacted Soils, together with a number of proposed revisions of existing standards.

Subcommittee G-2, Papers (W. G. Shockley, chairman) met in Washington, D. C., on January 10, 1961. At this meeting, offer forms for papers were reviewed and recommendations were made to Society headquarters for the presentation of five papers in the general soils session.

The committee recommends that all papers submitted for presentation at general soils sessions and specific symposia sessions be reviewed by Subcommittee G-2. In order to relieve the Subcommittee of some of the burden of review, a review panel is being assembled from D-18 membership for purposes of paper review only.

The Papers Subcommittee further recommends that reviews of papers be made available to the appropriate Society committees on prizes and awards when they are studying such papers for award purposes.

Subcommittee G-3—Nomenclature and Definitions (R. G. Ahlvin, chairman).—Organization of the subcommittee and joint committee (ASCE-ASTM) formation has accounted for nearly all of this year's activity. A meeting of Subcommittee G-3 and, insofar as possible, of the joint committee and of its individual elements is planned during the Annual Meeting in June. Specific coordination with Committee E-8 on Nomenclature and Definitions is being initiated.

Steering Committee on Research (C. B. Crawford, chairman) met once during the year on June 27, 1960 to consider a list of twenty-five problems of needed research which had been submitted to the Subcommittee. Each of these problems was assigned to one or more of the research subcommittees for study and action.

To promote the objectives of the Research Steering Committee, the Executive Committee decided to augment the membership by including all subcommittee chairmen on its membership.

Subcommittee R-1, Surface and Subsurface Reconnaissance (R. F. Baker, chairman).—The past year has been devoted to soliciting memberships and to defining the scope of the subcommittee's operations. Five new members have been added. Definite progress before and during the 1961 Annual Meeting is anticipated. No new or revised standards are pending.

Subcommittee R-2—Sampling and Related Field Testing for Soil Investigation (John P. Gnaedinger, chairman) developed the proposed Method for Core Drilling and has prepared revisions of Methods D 1586 – 58 T and D 1587 – 58 T.

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Vane shear testing procedures, dynamic cone penetration procedures, and use of nuclear methods for measuring in-place soil density and moisture contents are being studied.

Subcommittee R-3, Physical Characteristics of Soils (E. E. Bauer, chairman) recommended that tolerances be included in the following methods of test for the drying temperature: D 422, D 423, D 424, D 425, D 427, D 854, and, D 1140. Editorial revisions were recommended for D 427, D 1558, D 698, and D 1557. These proposed editorial revisions were approved by Committee D-18 at the 1960 Annual Meeting.

This subcommittee recommends revision of Method D 422 and D 423. Consideration is being given to the stirring paddle illustrated in Fig. 1 of Method D 422 to bring it up-to-date. Consideration of a new working drawing for the liquid limit device of Method D 423 is awaiting availability of the report on a study of all the devices currently being produced by the various manufacturers in the United States.

Consideration is being given to a method for calibrating mechanical equipment to be used in the compaction of test specimens in Methods D 698 and D 1557 and to the inclusion of mechanical equipment in both methods. Consideration is also being given to the preparation of a drawing for the hand rammers for these two methods. The subcommittee recommends that these methods and Method D 1558 remain as tentatives pending further consideration.

The work of Section D on Densities of Granular Soils has been interrupted by the death of Mr. Earl J. Felt, chairman of the section.

Subcommittee R-4, Physical Properties of Soils (N. W. McLeod, chairman).—At its meeting in June 1960, task forces were established to prepare a report on the specific objectives of Subcommittee R-4, and to assemble a list of important problems on the capillarity and permeability of soils on which research is urgently needed. A method for measuring the relationship between moisture content and capillary action of soils that have capillary potentials less than one atmosphere is being prepared as well as a method for measuring the permeability of granular soils. Subcommittee R-4 proposes holding a symposium on capillary phenomena associated with soils and base courses under highway and airport pavements, at the 1962 Annual Meeting.

Subcommittee R-5, Structural Properties of Soils—(D. M. Burmister, chairman).—A proposed tentative method of test for unconfined compression has been circulated to the subcommittee.

The subcommittee is studying the clarification of ideas and conceptions regarding angle of friction and cohesion of soils, as a basis for effective subcommittee work. There seems to be a growing general opinion that angle of friction and cohesion are not simple, constant single values and inherent properties of soils but that they are only "convenient parameters" for evaluating the results of shearing tests on soils, for expressing a complex set of soil properties which govern shearing failure of soils, and for making practical applications of the results of shearing tests on soils in stability investigations for earthworks, retaining structures, and foundations. The values of angle of friction and cohesion are strongly influenced and conditioned by the conditions that control, whether in the natural environment or modified by subsequent construction of structures or imposed in a shearing strength test. These basic aspects of shear testing of soils are to be explored and studied with regard to their implications in setting up testing procedures.

Subcommittee R-6, Physicochemical Properties of Soils (D. T. Davidson, chairman) continued to work with committees of the American Society of Agronomy responsible for preparing the Monograph "Methods of Soil Analysis," which is being cosponsored by ASTM without financial obligation. C. A. Black, Professor of Agronomy, Iowa State University of Science and Technology, is Editor-in-Chief of the Monograph. Authors have been obtained for all topics, and preparation of manuscripts is under way.

At its business meeting during the Annual Meeting in June, 1960, the subcommittee decided to prepare a tentative method for routine X-ray analysis of soils. It also was thought desirable to investigate the possibility for standards on other test methods for determining chemical or mineralogical properties of soils. Accordingly, Section A on X-Ray Identification of Soil Clays is presently being formed with R. L. Handy as chairman. An attempt will be made to reach a decision on other sections at the 1961 meeting.

Subcommittee R-8, Special and Construction Control Tests (B. S. Coffman, chairman) held a meeting during the 1960 Annual Meeting with all sections active.

Section A, Base Courses and Subgrades for Highways and Airports, prepared Method of Test for Determining the Bearing Ratio of Laboratory-Compacted Soils which is appended to this report. The section will study the promotion of other methods for bearing tests.

Section B, Earth Dams and Embankments, prepared a Method of Test for Determining the Density of Soil in Place by the Rubber Balloon Method which is receiving further study.

Section C-1, Cement Stabilization, is interested in a new method of testing soil-cement for cement content. Section C-2, Bituminous-Stabilization, has recommended to Subcommittee R-8 the adoption as standard of Method D 915 with an editorial revision of the scope.

It is recommended that the following remain as tentatives pending consideration by the Section for revision: D 916, D 1241, D 1411, D 1556, D 1632, D 1633, D 1634, and D 1635.

Subcommittee R-9, Dynamic Properties of Soils (R. K. Bernhard, chairman) is sponsoring the Symposium on Soil Dynamics to be presented during the Annual Meeting of the Society.

The Tentative Definitions of Terms Relating to Soil Dynamics have been accepted by the Administrative Committee on Standards.

Subcommittee R-11, Pile Load Bearing Tests (W. S. Housel, chairman) held a meeting on June 27, 1960 in conjunction with the Annual Meeting of the Society. During the year, revisions of Method D 1143 - 57 T were completed. The subcommittee has under consideration special problems in the area of pile testing. Initiation of the cooperative studies of these problems and in some cases preparation of suggested methods of tests are in progress. A suggested Method of Test for Load Capacity of Batter Pile Frames has been prepared and is now being revised preparatory to submission as a tentative method of test.

This report has been submitted to letter ballot of the committee, which consists of 116 voting members; 93 members returned their ballots, of whom 91 voted affirmatively, and 0 voted negatively.

Respectfully submitted on behalf of the committee,

W. G. HOLTZ,
Chairman.

ARTHUR A. WAGNER,
Secretary.

REPORT OF COMMITTEE D-19
ON
INDUSTRIAL WATER*

Committee D-19 on Industrial Water held meetings on June 29 to July 1, 1960, in Atlantic City, N. J., and on January 25 to 27, 1961, in Pittsburgh, Pa.

The Symposium on Impurities in Steam, sponsored by the committee, will be presented on June 29, 1961, in Atlantic City, N. J.[1] Included is a paper, "Research Report on Stoichiometry of the Vaporous Carry-over of Sodium Chloride from High-Pressure Boiler Water," presented on behalf of the committee by M. M. Rubright, Babcock & Wilcox Co., Research Center, Alliance, Ohio. This paper incorporates the results of the committee's research investigation on Vaporous Carry-over of Boiler Water Salts, financed by funds granted to the committee by the ASTM Administrative Committee on Research.

The committee recommended to the Society the reprinting of the Manual on Industrial Water and Industrial Waste Water, as *ASTM STP No. 148-E*, Second Printing of Second Edition, 1960. No changes were made in Part I, the text chapters. Part II includes all of the tentatives and standards prepared by Committee D-19 on Industrial Water, and published by the Society through 1960.

The ASTM representatives serving on the Joint Committee on Uniformity of Methods of Water Examination (JCUMWE) submitted to the Society and to the committee recommendations as follows, covering the period of July 1, 1960, to January 1, 1961:

Preliminary recommendations on sulfates, on uniformity of reagents, and on acidity, basicity, and alkalinity have not yet been voted on as official recommendations. The report on pH was returned to the panel for revision. ASTM comments were not ready for submission.

JCUMWE established new review panels on silica, chlorine residual, and chromium to which ASTM representatives are being appointed. Progress reports were received from the thirteen panels noted in the last report of JCUMWE activity.

Reports of activity of member organizations included the information that API has panels on ionized cyanide and arsenic.

C. K. Rice, Coordinator, Refinery Technological Laboratory of the Gulf Research and Development Co., Philadelphia, Pa. resigned from the committee, as Standards Advisor, and as one of the two ASTM representatives serving on JCUMWE. He was elected as an Honorary Member of the committee for the services rendered during the past eleven years.

A. C. Muller, of the Consolidated Edison Co. of New York, Inc., was appointed as the Standards Advisor of the committee, and Frank E. Clarke was appointed as the ASTM representative on JCUMWE.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] See p. 1369.

J. E. Kinney, Sanitary Engineering Consultant, was appointed as the ASTM Representative on the Engineers Joint Council, National Water Pollution Panel.

The 1961 Max Hecht Award Certificate and citation were presented on April 22, 1961, to Claude K. Rice.

F. E. Clarke, vice-chairman of the committee, was the recipient of one of the several ASTM Award of Merit Certificates on June 28, 1961, in Atlantic City, N. J.

Letters of condolences were sent to the families of the members as follows, deceased in 1960 and 1961: D. K. French; B. H. Ryan, representing the Deady Chemical Co.; and W. W. Hodge, of the Mellon Institute of Industrial Research.

The committee comprises 150 members, and operates with one advisory, eight technical subcommittees, and 78 task groups, distributed among the subcommittees. The committee's production of methods up to the present time includes 29 tentatives and 45 standards published by the Society.

H. J. Vyhnalek, Cleveland Electric Illuminating Co., and M. Daniels, Armco Steel Corp., were elected to serve as Members-at-Large on Subcommittee I, Advisory, for a three-year term starting June 30, 1961.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto:[2]

Tentative Methods of:

Test for Acidity and Alkalinity of Industrial Water (Subcommittee IV),
Test for Chloride Ion in High-Purity Industrial Water (Subcommittee IV),
Test for Nickel in Industrial Water (Subcommittee IV),
Test for Suspended and Dissolved Solids in Industrial Water (Subcommittee IV),
Test for Sodium in High-Purity Industrial Water by Flame Photometry (Subcommittee V),
Test for Turbidity of Industrial Water (Subcommittee V),
Test for Hexane-Extractable Matter in Industrial Waste Water (Subcommittee VII), and
Measurement of Beta Particle Radioactivity of Industrial Water and Industrial Waste Water (Subcommittee IX).

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Methods of Test for Iron in Industrial Water and Industrial Waste Water (D 1068 – 60 T)[3] (Subcommittee IV):

Section 15(b).—Change to read as follows:

> (*b*) The precision of the method varies with the quantity being determined as shown in Fig. 1.
>
> The accuracy of the method is +1 per cent at the 0.02-mg iron level and −0.5 per cent at the 0.4-mg iron level.

Add a new Fig. 1 as shown in the accompanying Fig. 1.[4]

Tentative Method of Test for Silica in High-Purity Water (D 1689 – 59 T)[5] (Subcommittee IV):

New Table.—Add a new Table I as shown in the accompanying Table I.

Note 2.—Change to read as follows:

> NOTE 2.—If the photometer is not capable of measuring absorbance at 815 mμ, measurements may be made in the range 640 mμ to 700 mμ (Table I). However, measurements made at 815 mμ provide maximum sensitivity and reproducibility.

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[2] The new tentatives appear in the 1961 Book of ASTM Standards, Part 10.

[3] 1960 Supplement to Book of ASTM Standards, Part 10.

[4] Not included here; see the revised method, 1961 Book of ASTM Standards, Part 10.

[5] 1959 Supplement to Book of ASTM Standards, Part 10.

Section 12.—Change to read as follows:

12. (*a*) The precision of this method varies with the quantity being determined as shown in Fig. 1. The longer the cell or scale length, or both, consistent with an acceptable transmittance range, the better the precision obtainable (Note 5).

NOTE 5.—The data in Fig. 1 were obtained using both filter photometers and spectrophotometers. With a 100-mm cell length and a spectrophotometer, better precision is obtainable.

TABLE I.—APPLICATION RANGES OF APPARATUS FOR MEASUREMENT OF COLOR.

(New Table I, Method D 1689)

Apparatus for Color Measurement	Range Covered,[a] ppb
Spectrophotometer (815 $m\mu$):[b]	
10-mm cell	50 to 1000
100-mm cell	2 to 200
Filter photometer (red filter):[b, c]	
23-mm cell	20 to 1000
50-mm cell	10 to 1000

[a] For the usual 50-ml sample.

[b] A discussion of photometers and photometric practice is given in the Recommended Practice for Photometric Methods for Chemical Analysis of Metals (ASTM Designation: E 60), 1960 Book of ASTM Methods for Chemical Analysis of Metals.

[c] A Fisher No. 650 filter or a Klett-Summerson No. 66 filter, or the equivalent, has been found satisfactory for this purpose.

(*b*) The accuracy of this method is estimated to be within +0.5 ppb at a concentration of about 5 ppb, and −0.9 per cent of the accepted value at concentrations approaching 1000 ppb.

Add a new Fig. 1 as shown in the accompanying Fig. 2.[4]

Tentative Method of Test for Odor in Industrial Water and Industrial Waste Water (D 1292 – 59 T)[5] (Subcommittee VII):

Section 1.—Delete Note 1 under Paragraph (*a*).

Section 3.—Immediately preceding the definition of "threshold odor number," insert the definition of "odor intensity index," to read as follows:

Odor Intensity Index.—The number of times the concentration of the original sample is halved by addition of odor-free water to obtain the least definitely perceptible odor.

Change the definition of threshold odor number to read "The greatest dilution of the sample with odor-free water to yield the least definitely perceptible odor."

Section 6(*b*).—After the third sentence, add the following two sentences: "Some plastic tubing may also cause odor. An all glass apparatus is ideal."

Section 9.—Change the ninth and tenth sentences of Paragraph (*b*) to read as follows:

The odor tester shall select the flask that has a perceptible odor. If the odor tester fails to detect an odor, the dilution tester then shall decrease the dilution (increasing the concentration) until a dilution is found at which the odor is perceptible using the same procedure.

In the first sentence of Paragraph (*c*), delete the word "immediate." Change the second sentence of Paragraph (*c*) to read: "Repeat this procedure until the odor tester fails to detect an odor."

Section 10.—Change Paragraphs (*a*) and (*b*) to read as follows:

10. (*a*) Calculate the odor intensity as threshold odor number as follows:

$$\text{Threshold odor number} = \frac{200}{A} \times 8^{D}$$

where:

A = milliliters of sample or milliliters of aliquot of the primary dilution used, and

D = number of 25:175 primary dilutions required to reach the determinable magnitude of odor intensity.

(*b*) Calculate the odor intensity as odor intensity index as follows:

$$\text{Odor intensity index} = 3.3 \log \frac{200}{A} + 3\,D$$

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Scheme for Analysis of Industrial Water and Industrial Waste Water (**D 1256 – 59 T**)[5] (Subcommittee II) be approved for reference to letter ballot of the Society for adoption as standard without revision.

Adoption of Tentatives as Standard With Revision

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

Tentative Methods of Test for Sodium and Potassium Ions in Industrial Water and Water-Formed Deposits by Flame Photometry (D 1428 – 56 T)[6] (Subcommittee V):

Section 1.—Replace the present last two sentences of Paragraph (*a*) with the following:

The methods are described as follows:

	Sections
Method A. Analysis of Industrial Water	8 to 13
Method B. Analysis of Water-Formed Deposits	14 to 19

Add the following as a new first sentence in Paragraph (*b*):

The determination of sodium in high-purity industrial water is covered in the Method of Test for Sodium in High-Purity Industrial Water by Flame Photometry (ASTM Designation: D 1887).

Section 8.—Add the following sentence at the end of this section: "For samples containing less than 1 ppm sodium, Method D 1887 for determining sodium in high-purity industrial water is recommended."

Section 13.—Change to read as follows:

13. The precision of this method is ±2.0 per cent for sodium and ±5.0 per cent for potassium.

Sections 14 to 19.—Delete these sections, which cover the present Method B. Redesignate the present Method C as Method B and renumber the present Sections 20 to 25 as Sections 14 to 19.

Section 23.—Following the sixth sentence, add the following new sentence: "Repeat the digestion and ignition until the solution appears clear."

Section 25.—Renumber as Section 19 and change to read as follows:

19. The precision of this method is ±5.0 per cent for sodium and ±9.0 per cent for potassium.

Tentative Methods of Test for Total Chromium in Industrial Water and Industrial Waste Water (D 1687 – 59 T)[5] (Subcommittee VII):

Section 2(*b*).—Change the last two sentences to read as follows:

In general, Method A is applicable where organic matter is low or easily oxidized. Method B is applicable in the presence of large amounts of refractory organic matter, as in tannery wastes.

Section 4.—Change Paragraph (*a*) to read as follows:

4. (*a*) To develop the color quantitatively, all the chromium must be converted to the hexavalent condition and any organic matter destroyed. The color reaction for chromium is quite specific. With Method A, iron in excess of 1000 ppm, vanadium in excess of 4 ppm, and molybdenum and mercury in amounts over 200 ppm may interfere. For Method B, iron in excess of 50 ppm and other precipitable hydroxides may interfere. Hence, these elements, if present in excess of the above quantities, must be removed or converted to a noninterfering complex.

In the second line of Paragraph (*b*), change "200" to read "250."

Add a new Paragraph (*c*) to read as follows:

(*c*) Large amounts of phosphate and alkali earth metal may cause precipitation and loss of chromium during the alkaline oxidation of Method B. If this occurs, an acid oxidation, as in Method A, should be used.

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[6] 1958 Book of ASTM Standards, Part 10.

Reletter the present Paragraph (*c*) as (*d*).

Note 2.—Renumber as Note 3 and transfer to follow Section 9(*f*).

Note 3.—Renumber as Note 2.

Section 9(*i*).—Change "75" to '49" in the first and third lines of this paragraph.

Note 6.—Change "20 ml of H_2SO_4 (1:75)" to read "13 ml of H_2SO_4 (1:49)."

Section 11(*c*).—Change to read as follows:

(*c*) Cool the solution to room temperature. Add 17 ml of water and neutralize with NH_4OH (1:1). Add 13 ml of H_2SO_4 (1:49). Swirl and, if necessary, warm to achieve solution.

Section 16(*h*).—In lines 1 and 4, change "7" to "9."

Section 18(*d*).—Change the first sentence to read: "From a microburet, add 1.16 ml of H_2SO_4 (1:9)."

Tentative Method for Measurement of Gamma Radioactivity of Industrial Water and Industrial Waste Water (D 1690 - 59 T)[5] (Subcommittee IX):

Section 3.—Delete from Paragraph (*c*) the words "micromicrocurie or," and change "3.7" to "3.700" and "2.22" to "2.220."

Change Paragraph (*d*) and add a new Note 1 to read as follows:

(*d*) *Counter Background.*—In the measurement of radioactivity, the counting rate resulting from factors other than the radioactivity of the sample and reagents used (Note 1).

NOTE 1.—Counter background varies with the location, shielding of the detector, and the electronics; it includes cosmic rays, contaminating radioactivity, and electrical noise.

Change Paragraph (*e*) to read as follows:

(*e*) *Radioactively Homogeneous Water.*—Water in which the radioactive material is uniformly dispersed throughout the volume of water sample and remains so until the measurement is completed, or until the sample is evaporated or precipitating reagents are added to the sample.

Delete the present Note 1.

Section 8.—Change Paragraph (*b*) and add a new Note 4 to read as follows:

(*b*) Preserve the sample in a radioactively homogeneous state (Note 4).

NOTE 4.—A sample may be made radioactively homogeneous by addition of a reagent in which the radioelements or compounds of the radioelements present in the sample would be soluble in large concentrations. Addition of acids, complexing agents, or like stable carriers may be used to obtain homogeneity. Consideration of the chemical nature of the radioelements and compounds present and the subsequent chemistry of the method will indicate the action to be taken.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Definitions of Terms Relating to Industrial Water and Industrial Waste Water[3] (D 1129 - 60) (Subcommittee II):

Delete the existing definition for "Background."

Revise existing definitions to read as follows:

Curie.—A unit of radioactivity equivalent to 3.700×10^{10} atomic disintegrations per second or 2.220×10^{12} atomic disintegrations per minute. A microcurie is 1 one-millionth of a curie (10^{-6} curie); a picocurie, 1 one-millionth of a microcurie (10^{-12} curie). (D 1690 and D 1890)

Dissolved Solids (Dissolved Matter).—The dried residue from evaporation of the filtrate after separation of suspended solids (suspended matter). (D 1069)

Radioactively Homogeneous Water.—Water in which the radioactive material is uniformly dispersed throughout the volume of water sample and remains so until the measurement is completed or until the sample is evaporated or precipitating reagents are added to the sample. (D 1690 and D 1890)

Suspended Solids (Suspended Matter).—Those solids (that matter) which can be separated from the sample by filtration. (D 1069)

Total Solids (Total Matter).—The sum of the suspended and dissolved solids (matter). (D 1069)

New Definitions.—Add the following new definitions in proper alphabetical sequence:

Biological-Chemical Oxygen Demand.—The quantity of oxygen required for the biological and chemical oxidation of water-borne substances.

Counter Background.—In the measurement of radioactivity, the counting rate resulting from factors other than the radioactivity of the sample and reagents used. (D 1690 and D 1890)

NOTE.—Counter background varies with the location, shielding of the detector, and the electronics; it includes cosmic rays, contaminating radioactivity, and electrical noise.

Counter Beta-Particle Efficiency.—In the measurement of radioactivity, that fraction of beta particles emitted by a source which is detected by the counter. (D 1890)

Maximum Beta Energy.—The maximum energy of the beta-particle energy spectrum produced during beta decay of a given radioactive species.

NOTE.—Since a given beta-particle emitter may decay to several different quantum states of the product nucleus, more than one maximum energy may be listed for a given radioactive species.

Reagent Background.—In the measurement of radioactivity of water samples, the counting rate observed when a sample is replaced by mock sample salts or by reagent chemicals used for chemical separations.

NOTE.—Reagent background varies with the reagent chemicals and analytical methods and may vary with reagents from different manufacturers and from different processing lots.

Standard Methods of Test for Fluoride Ion in Industrial Water and Industrial Waste Water (D 1179 – 58)[6] (Subcommittee IV):

Footnote 1.—Change the second paragraph to read as follows:

These methods have been adapted from analytical methods developed by the Branch of Quality of Water, U. S. Geological Survey, as published in "Methods for Collection and Analysis of Water Samples," U. S. Geological Survey Water-Supply Paper 1454, pp. 163–171 (1960). For a more thorough discussion, see W. L. Lamar and P. G. Drake, "Factors Affecting the Determination of Fluoride in Water with Zirconium-Alizarin," *Journal*, Am. Water Works Assn., Vol. 47, June, 1955, pp. 563–572, and L. L. Thatcher, "Modified Zirconium-Eriochrome Cyanine R Determination of Fluoride," *Analytical Chemistry*, Vol. 29, November, 1957, pp. 1709–1712.

Footnote 5.—Delete the first paragraph.

Section 10.—Delete Paragraphs (*a*) and (*b*) and Note 2 and replace with the following new Paragraphs (*a*) to (*c*) and new Note 2, renumbering the present Paragraphs (*c*) to (*e*) as Paragraphs (*d*) to (*f*).

10. (*a*) Dissolve in 50 ml of H_2SO_4, by stirring, sufficient Ag_2SO_4 to precipitate the chloride present in the sample. A slight excess of Ag_2SO_4 will do no harm.

(*b*) Transfer 200 ml of the sample (Note 2) to the fluoride distillation flask, make alkaline to phenolphthalein with a pellet of NaOH, add some glass beads, and evaporate to a volume of about 50 ml, preferably using an electrical heating mantle. Care must be exercised to prevent bumping and consequent loss of sample.

NOTE 2.—If 200 ml of the sample contain more than 4000 mg of chloride, take a smaller aliquot so that the chloride in the sample aliquot will not exceed 4000 mg chloride.

(*c*) Allow the concentrated sample to cool and make slightly acid by dropwise addition of H_2SO_4. Connect the distillation assembly except for the steam inlet tube, *K* (Fig. 1). In place of the steam inlet tube insert a long-stem cylindrical separatory funnel. Start a flow of cooling water through the condenser. Close the stopcock on the separatory funnel, and introduce into the funnel 50 ml of the H_2SO_4 - Ag_2SO_4 solution. Slowly add the H_2SO_4 - Ag_2SO_4 solution to the fluoride distillation flask, regulating the addition of the solution by means of the stopcock on the separatory funnel. Tilt or shake flask *M* to thoroughly mix the H_2SO_4 - Ag_2SO_4 solution with the sample.

Section 13(i).—In the next to last sentence, change "H_2SO_4 (1:17)" to read "H_2SO_4 (2.1 *N*)."

Section 15.—Change Paragraph (*d*) to read as follows:

> (*d*) *Sulfuric Acid* (2.1 *N*).—Dilute 29.1 ml of concentrated sulfuric acid (H_2SO_4, sp gr 1.84) to 500 ml by carefully adding the H_2SO_4 to the water.

In Paragraph (*e*), change "H_2SO_4 (1:17)" to "H_2SO_4 (2.1 *N*)."

Section 21.—In Paragraph (*a*), in place of "lead, cadmium, and phosphate, 5 ppm" substitute "lead and cadmium, 5 ppm; metaphosphate (PO_3), 1.0 ppm, and orthophosphate (PO_4), 10 ppm."

Delete Paragraph (*f*).

Note 12.—Change to read as follows:

> NOTE 12.—At the time of reading, all solutions should be at the same temperature and within 3 C of the temperature at which the calibration curve was prepared. Temperature variations may occur if the reference solution is allowed to remain in the spectrophotometer during prolonged testing.

WITHDRAWAL OF TENTATIVE AND STANDARD

The committee recommends the withdrawal of the following methods:

Tentative Methods of Test for:

Acidity and Basicity (Alkalinity) in Industrial Water and Industrial Waste Water (**D 1067 - 57 T**) (Subcommittees IV and VII).

Standard Methods of Test for:

Suspended and Dissolved Matter (Suspended and Dissolved Solids) in Industrial Water and Industrial Waste Water (**D 1069 - 58**) (Subcommittees IV and VII).

New methods for the above determinations in industrial waste water are now in preparation. New methods for these determinations in industrial water other than waste water are covered elsewhere in this report.

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Methods of Test for:

Total Aluminum and Aluminum Ion in Industrial Water (**D 857 - 50**) (Subcommittee IV),
Iodide and Bromide Ions in Industrial Water (**D 1246 - 55**) (Subcommittee IV),
Calcium Ion and Magnesium Ion in Industrial Water (**D 511 - 52**) (Subcommittee IV),
Hydroxide Ion in Industrial Water and Industrial Waste Water (**D 514 - 47**) (Subcommittee IV),
Manganese in Industrial Water and Industrial Waste Water (**D 858 - 49**) (Subcommittee IV), and
Nitrate Ion in Industrial Water (**D 992 - 52**) (Subcommittee IV).

Methods of:

Reporting Results of Examination and Analysis of Water-Formed Deposits (**D 933 - 50**) (Subcommittee II),
Field Sampling of Water-Formed Deposits (**D 887 - 49**) (Subcommittee III),
Sampling Water from Boilers (**D 860 - 54**) (Subcommittee III),
Examination of Water-Formed Deposits by Chemical Microscopy (**D 1245 - 55**) (Subcommittee V),
Identification of Crystalline Compounds in Water-Formed Deposits by X-Ray Diffraction (**D 934 - 52**) (Subcommittee V),
Corrosivity Test of Industrial Water (NDHA Method) (**D 935 - 49**) (Subcommittee VI), and
Corrosivity Test of Industrial Water (USBM Embrittlement Detector Method) (**D 807 - 52**) (Subcommittee VI).

Specification for:

Substitute Ocean Water (**D 1141 - 52**) (Subcommittee VI).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision in substance of the following tentatives:

Tentative Methods of:

Sampling Homogeneous Industrial Waste Water (**D [illegible]6 - 57 T**) (Subcommittee III), Sampling Industrial Water (**D 510 – 55 T**) (Subcommittee III), and

Sampling Steam (**D 1066 – 59 T**) (Subcommittee III).

Tentative Specifications for:

Equipment for Sampling Industrial Water and Steam (**D 1192 – 51 T**) (Subcommittee III).

Tentative Methods of Test for:

Chloride Ion in Industrial Water and Industrial Waste Water (**D 512 – 55 T**) (Subcommittee IV),

Copper in High-Purity Water (**D 1688 – 59 T**) (Subcommittee IV),

Dissolved Oxygen in Industrial Water (**D 888 – 49 T**) (Subcommittee IV),

Hydrazine in Industrial Water (**D 1385 – 58 T**) (Subcommittee IV),

Iron in High-Purity Industrial Water (**D 1497 – 57 T**) (Subcommittee IV),

Silica in Industrial Water and Industrial Waste Water (**D 859 – 55 T**) (Subcommittee IV),

Sodium and Potassium in Industrial Water (**D 1127 – 50 T**) (Subcommittee IV),

Sulfate Ion in Industrial Water and Industrial Waste Water (**D 516 – 59 T**) (Subcommittee IV),

Sulfite Ion in Industrial Water (**D 1339 – 54 T**) (Subcommittee IV),

Zinc in Industrial Water and Industrial Waste Water (**D 1691 – 59 T**) (Subcommittee IV),

Electrical Conductivity of Industrial Water and Industrial Waste Water (**D 1125 – 59 T**) (Subcommittee V), and

Effect of Water in Tubular Heat Exchangers (**D 1591 – 58 T**) (Subcommittee VI).

EDITORIAL CHANGES

The committee recommends editorial changes as follows in the Standard Method of Field Sampling of Water-Formed Deposits (**D 887 – 49**)[6] (Subcommittee III):

Section 2.—Add a new Paragraph (*f*) to read as follows:

> (*f*) For definitions of other terms used in this method, refer to the Definitions of Terms Relating to Industrial Water and Industrial Waste Water (ASTM Designation: D 1129).

Note 23.—Change to read as follows: "A wide-mouth glass bottle, plastic vial, or noncorrodible metal container will provide adequate protection for samples of deposits."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[7]

This report has been submitted to letter ballot of the committee, which consists of 150 members; 82 members returned their ballots, of whom 70 have voted affirmatively and 3 negatively.

Respectfully submitted on behalf of the committee,

MAX HECHT,
Chairman.

ORRIN M. ELLIOTT,
Secretary.

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[7] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE D-20
ON
PLASTICS*

Committee D-20 on Plastics held three meetings during the past year: in Atlantic City, N. J., on June 28 to July 1, 1960, at the Annual Meeting of the Society; in Detroit, Mich., Oct. 3 to 6, 1960; and in Louisville, Ky., March 7 to 10, 1961.

The committee consists of 538 individuals representing 263 voting members, of whom 133 are classed as producers, 65 as consumers, and 65 as general interest members.

Committee D-20 has adopted as policy, the development of standards for plastic products. These standards are to be limited to pertinent performance properties and test methods, with design features specifically excluded.

A joint D-11/D-20 Task Group on Definitions has been established and has met twice to coordinate definitions of terms of mutual interest. The joint committee has recommended expansion of its scope to coordinate all matters of mutual interest between the two committees.

Representatives of Committees D-20 and C-16 on Thermal Insulating Materials met on January 31, 1961, in Cincinnati, Ohio, during Committee Week to discuss jurisdictional and technical problems associated with test methods and specifications for cellular plastic types of thermal insulation.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1960 Annual Meeting, Committee D-20 presented to the Society through the Administrative Committee on Standards the recommendations listed below. The recommendations were accepted by the Administrative Committee on the dates indicated.

New Tentative Methods of Test for:

Transparency of Plastic Sheeting (D 1746 - 60 T) (Approved Sept. 29, 1960),

Tensile Impact Energy to Break Plastics and Electrical Insulating Materials (D 1822 - 61 T) (Approved Jan. 11, 1961),

Apparent Viscosity of Plastisols and Organosols at Low Shear Rates by Brookfield Viscometer (D 1824 - 61 T) (Approved March 1, 1961), and

Apparent Viscosity of Plastisols and Organosols at High Shear Rates by Castor-Severs Viscometer (D 1823 - 61 T) (Approved March 1, 1961).

New Tentative Specifications for:

Poly(Vinyl Chloride) Resins (D 1755 - 60 T) (Approved Sept. 29, 1960), and

Epoxy Resins (D 1763 - 60 T) (Approved Sept. 29, 1960).

Revision of Tentative Specification for:

Polyethylene Molding and Extrusion Materials (D 1248 - 60 T) (Approved September 29, 1960).

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

Revision of Tentative Recommended Practice for:

Exposure of Polymeric Materials to High Energy Radiation (D 1672 – 59 T) (Jointly with Committee D-9) (Approved May 10, 1961).

Revision of Tentative Methods of Test for:

Resistance of Plastics to Chemical Reagents (D 543 – 60 T) (Approved Sept. 29, 1960), and

Carbon Black in Ethylene Plastics (D 1603 – 60 T) (Approved Sept. 29, 1960).

Revision and Reversion to Tentative of Standard Methods of Test for:

Specific Gravity of Plastics (D 792 – 60 T) (Approved Sept. 29, 1960), and

Ethyl Cellulose Molding and Extrusion Compounds (D 787 – 61 T) (Approved March 1, 1961).

Revision of Tentative Definitions of Terms Relating to:

Plastics (D 883 – 60 T) (Approved Sept. 29, 1960).

Those recommendations which were accepted by the Standards Committee on September 29, 1960 appear in the 1960 Supplement to Book of ASTM Standards, Part 9. The other items are available as separate reprints.

Committee D-20 had also submitted to the Standards Committee a proposed new Recommended Practice for Determining Light Dosage in Light and Weathering Apparatus. Approval has been withheld pending the availability of standard fading chips which are a requirement in the recommended practice.

NEW TENTATIVES

The committee recommends for publication as tentative the following specifications, methods of test, and recommended practices as appended hereto:[1]

Tentative Specification for:

Styrene-Butadiene Molding and Extrusion Materials.

Tentative Methods of Test for:

Blocking of Plastic Film,
Coefficients of Friction of Plastic Film, and
Apparent Density, Bulk Factor, and Pourability of Plastic Materials.

Tentative Recommended Practices for:

Transfer Molding of Thermosetting Materials,
Injection Molding of Specimens of Polystyrene Molding and Extrusion Materials, and
Sampling of Plastics.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Specification for Cellulose Acetate Molding and Extrusion Compounds (D 706 – 55 T):[2] Revise as appended hereto.[1]

Tentative Specification for Cellulose Acetate Butyrate Molding and Extrusion Compounds (D 707 – 55 T):[2] Revise as appended hereto.[1]

Tentative Specification for Nylon Injection Molding and Extrusion Materials (D 789 – 59 T):[3]

Section 3(*c*).—Change to read as follows:

> The moisture content of the resins described in this specification shall not exceed 0.28 per cent as received from the vendor as determined by the test method outlined in Section 6(*h*) of this specification (Note 2).

Section 6(*h*).—Add a new Section 6(*h*) including Fig. 1 (as shown in the accompanying Fig. 1[4]) to read as follows:

> (*h*) *Moisture Content.*—The moisture content of nylon materials shall be determined as follows:
>
> (*1*) *Apparatus.*—A heating device capable of heating the test tubes to the temperature specified in Item (*4*) (*c*) of this section (electrically heated aluminum block, hot oil or molten salt bath, or hot air oven is acceptable); vacuum

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[1] The new and revised tentatives appear in the 1961 Book of ASTM Standards, Part 9.

[2] 1958 Book of ASTM Standards, Part 9.

[3] 1959 Supplement to Book of ASTM Standards, Part 9.

[4] Not included here; see revised tentative, 1961 Book of ASTM Standards, Part 9.

source capable of maintaining a pressure of 4 mm Hg absolute or less; mercury U tube or McLeod Gage; vapor traps equivalent to Scientific Glass Apparatus Co. No. J-4310; Dewar flask or equivalent to maintain vapor trap below minus 70 C; thermometers; 25 mm by 200 mm test tubes (Pyrex or equivalent); No. 5 solid neoprene stopper; eye dropper with fine tip; glass or stainless steel tubing of 5 mm minimum inside diameter; 25 ml pipet; 125 ml Erlenmeyer flask with glass stopper; squeeze bulb; iodine crystals; desiccator or drying oven; anhydrous methanol; analytical balance accurate to 1.0 mg.

(*2*) *Preparation and Standardization of Karl Fischer Reagent:*

(*a*) Mix thoroughly a 1:1 solution of Karl Fischer Reagent (strength of reagent before dilution to be such that 1 ml of solution is equivalent to 6 mg of water) and methanol.

(*b*) Weigh a dropper of distilled water, introduce two small drops of the water into a previously dried glass-stoppered Erlenmeyer flask and stopper immediately. Reweigh dropper and record weight of water in flask.

(*c*) Make up a standard color solution from 0.0636 g iodine in 25 ml of methanol.

(*d*) Titrate the water in the flask with the diluted Karl Fischer Reagent to an end point given by the standard color solution. Record the titer (Note 10).

(*e*) Calculate the Karl Fischer Equivalent Number (KFE) as follows:

$$\text{KFE} = \frac{\text{H}_2\text{O Weight}}{\text{ml of KFR (diluted)}}$$

(*3*) *Sample Preparation.*—Samples for moisture determination should be retained in glass vials with plastic caps having aluminum foil paper board liners taped in place after tight closure using two turns of vinyl electrical tape. Vials, with aluminum foil-lined cap, 107 mm by 28 mm diameter, will hold 20 to 30 g of polymer (depending upon size and shape of chip). Weighing errors can easily occur if delays are incurred during the weighing of samples for test.

(*4*) *Moisture Determination:*

(*a*) Weigh sample and vial (removing tape immediately before weighing). Quickly transfer sample from vial to the previously dried test tube to be used for moisture determination and immediately stopper the test tube. Reweigh empty vial and cap to determine the weight of sample used for this determination.

(*b*) Assemble previously dried cold trap and test tube with sample and start vacuum pump (See Fig. 1). Evacuate for 2 min then pre-cool cold trap in a beaker of solvent and dry ice for about 15 sec. Remove beaker and insert cold trap in Dewar flask containing solvent and dry ice. Maintain a maximum pressure of 4 mm Hg absolute and a temperature of minus 70 C or lower in the cold trap (Dewar flask).

(*c*) Immerse the test tube containing the moisture sample in the heat bath as follows:

Type I, II, III, V, VI and VII Resins, 30 min at 260 C ± 2 C

Type IV Resin, 45 min at 180 C ± 2 C

(*d*) Release the vacuum (Note 7) and remove the cold trap from the cold bath and wash down the cold trap with 25 ml methanol (Note 8). Restopper the cold trap.

(*e*) Wait 15 min before titrating the solution from the cold trap (Note 9). Titrate the solution in the cold trap with the dilute standardized KFR to the same end point as for the KFE. Run a blank test tube exactly as in Paragraph (*4*) (*b*), (*c*), (*d*), and (*e*) and subtract from ml used by the sample. Record the difference as ml of KFR (Note 10).

$$\text{Water, per cent} = \frac{\text{ml KFR} \times \text{KFE} \times 100}{\text{weight of sample}}$$

(*5*) *Accuracy.*—The accuracy of this method has been shown to be ±0.05 per cent moisture at the 95 per cent confidence level using the average of single determinations from each of two samples, when the moisture content of the resin does not exceed 0.28 per cent.

Footnote 3.—Delete the present Footnote 3, renumbering subsequent footnotes accordingly.

Notes.—Delete the last sentence of Note 1.

Add new Notes 7, 8, 9, and 10 to read as follows:

NOTE 7.—A procedure for releasing the vacuum in the system with dry CO_2 is used by some laboratories to avoid introducing moisture into the system at this step.

NOTE 8.—This amount of washdown will permit positive transfer of condensed moisture and permit the option to titrate two 10-ml aliquots if desired but an appropriate correction factor must be applied to the formula (*4*) (*e*) of Section (6)(*h*) when less than the total solution is titrated.

NOTE 9.—It has been observed that sometimes minute ice crystals form in the cold trap and this delay period allows time for the trap to warm up and melt those crystals.

NOTE 10.—The containers used for the color standard and for the titrations should be identical and the volume of solution in each should be approximately equal at the end of the titration.

Tentative Method of Test for Stiffness in Flexure of Plastics (D 747 - 58 T):[2]

Revise as appended hereto.[1]

Tentative Methods of Testing Urethane Foam Raw Materials (D 1638 - 60 T):[5]

Revise as appended hereto.[1]

Tentative Definitions of Terms Relating to Plastics (D 883 - 60 T):[5]

New Definitions.—Add the following new definitions, inserting them in the proper alphabetical sequence:

Alkyd Plastics.—Plastics based on resins composed principally of polymeric esters, in which the recurring ester groups are an integral part of the main polymer chain, and in which ester groups occur in most crosslinks that may be present between chains.

Cast Film.—A film made by depositing a layer of plastic, either molten, in solution, or in a dispersion, onto a surface, stabilizing this form, and removing the film from the surface.

Gusset, ***n.***—(*1*) A piece used to give additional size or strength in a particular location of an object.

(*2*) The folded-in portion of flattened tubular film.

Necking, ***n.***—The localized reduction in cross-section which may occur in a material under tensile stress.

TENTATIVE REVISION OF STANDARD

The committee recommends tentative revision as follows of the Standard Specifications for Molds for Test Specimens of Plastic Molding Materials **(D 617 - 57):**[2]

New Section 7.—Add a new Section 7, including the footnote and Fig. 8 (as shown in the accompanying Fig. 2),[6] to read as follows:

Mold for Transfer Molding of Thermosetting Test Specimens

7. (*a*) *Mold.*—The cavities of this mold are designed to mold in one shot the following test specimens: a 6.4 by 12.7 by 127-mm ($\frac{1}{4}$ by $\frac{1}{2}$ by 5-in.) bar; a 12.7 by 12.7 by 127-mm ($\frac{1}{2}$ by $\frac{1}{2}$ by 5-in.) bar; a 3.2 by 50.8-mm ($\frac{1}{8}$ by 2-in.) diameter disk; a 3.2 by 102-mm ($\frac{1}{8}$ by 4-in.) diameter disk; and a 3.2 by 216-mm ($\frac{1}{8}$ by $8\frac{1}{2}$-in.) tension test specimen. The mold for these specimens shall be of the design shown in Fig. 8[a] and shall conform to the requirements prescribed in the following Paragraphs (*b*) to (*e*).

(*b*) *Gate Size.*—The gate size shall have a rectangular cross-section 6.4 mm wide by 1.52 mm deep ($\frac{1}{4}$ by 0.060 in.).

(*c*) *Mold for Tension Specimen.*—The cavity of the mold for tension specimens for the Method of Test for Tensile Properties of Plastics (ASTM Designation: D 638) shall have dimensions for the type 1 specimen as shown in that method except for the following thickness:

$$3.17\ \text{mm} \left\{\begin{matrix}+0.076\\-0.000\end{matrix}\right. \left(0.125\ \text{in.} \left\{\begin{matrix}+0.003\\-0.000\end{matrix}\right.\right)$$

(*d*) *Mold for Bar Specimens.*—The cavities of the mold for the bar specimens shall have the following tolerances:

Nominal Cavity Dimension	Specimen 12.7 by 12.7 mm (1/2 by 1/2 in.)	Specimen 6.4 by 12.7 mm (1/4 by 1/2 in.)
12.7 mm......	$12.70\left\{\begin{matrix}+0.127\\-0.000\end{matrix}\right.$	$12.70\left\{\begin{matrix}+0.127\\-0.000\end{matrix}\right.$
(½ in.).......	$\left(-0.500\left\{\begin{matrix}+0.005\\-0.000\end{matrix}\right.\right)$	$\left(-0.500\left\{\begin{matrix}+0.005\\-0.000\end{matrix}\right.\right)$
6.4 mm.......	...	$6.35\left\{\begin{matrix}+0.076\\-0.000\end{matrix}\right.$
(¼ in.)........	...	$\left(-0.250\left\{\begin{matrix}+0.003\\-0.000\end{matrix}\right.\right)$
Length........	12.7 cm (5 in.)	12.7 cm (5 in.)

(*e*) *Mold for Disk Specimens.*—The cavities of the mold for the disk specimens shall have the following dimensions:

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[5] 1960 Supplement to Book of ASTM Standards, Part 9.

[6] Not included here; see tentative revision of Method D 647, 1961 Book of ASTM Standards, Part 11.

Maximum Diameter.	50.8 mm (2 in.)	102 mm (4 in.)
Depth......	3.175 mm $\left\{\begin{matrix}+0.076\\-0.000\end{matrix}\right.$ $\left(-0.125 \text{ in.} \left\{\begin{matrix}+0.003\\-0.000\end{matrix}\right.\right)$	3.175 mm $\left\{\begin{matrix}+0.076\\-0.000\end{matrix}\right.$ $\left(-0.125 \text{ in.} \left\{\begin{matrix}+0.003\\-0.000\end{matrix}\right.\right)$
Edge Taper (to bottom of cavity)...	10 deg	10 deg

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[a] Blueprint of detailed drawings for the construction of the mold shown in Fig. 8 is available at nominal cost from the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends revisions as indicated of the following standards and their reversion to tentative:

Standard Method of Test for Stiffness Properties of Nonrigid Plastics as a Function of Temperature by Means of a Torsional Test (D 1043 – 51):[2] Revise as appended hereto.[1]

Standard Recommended Practice for Molding Test Specimens of Phenolic Materials (D 796 – 51):[2] Revise as appended hereto.[1]

REVERSION OF STANDARD TO TENTATIVE WITHOUT REVISION

The committee recommends that the following standards be reverted to tentative without revision:

Standard Methods of:

Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (D 392 – 38),[2]
Test for Compressive Properties of Rigid Plastics (D 695–54),[2] and
Test for Luminous Reflectance, Transmittance, and Color of Materials (D 791 – 54).[2]

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods of Test for:

Flammability of Plastics 0.050 in. and Under in Thickness (**D 568 – 56 T**),[2]
Flexural Properties of Plastics (**D 790 – 59 T**),[3]
Ammonia in Phenol-Formaldehyde Molded Materials (**D 834 – 59 T**),[3]
Haze and Luminous Transmittance of Transparent Plastics (**D 1003 – 59 T**),[3]
Tear Resistance of Plastic Film and Sheeting (**D 1004 – 59 T**),[3]
Orientation Release Stress of Plastic Sheeting (**D 1504 – 57 T**),[3]
Dilute Solution Viscosity of Ethylene Polymers (**D 1601 – 59 T**),[3]
Tensile Heat Distortion Temperature of Plastic Sheeting (**D 1637 – 59 T**),[3]
Indentation Hardness of Plastics by Means of a Durometer (**D 1706 – 59 T**),[3] and
Particle Size Analysis of Powdered Polymers and Copolymers of Vinyl Chloride (**D 1705 – 59 T**).[3]

Tentative Specifications for:

Extruded Acrylic Plastic Sheet (**D 1547 – 59 T**),[3] and
Nonrigid Vinyl Chloride Plastic Sheeting (**D 1593 – 58 T**).[2]

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee, jointly with Committee D-9 on Electrical Insulating Materials, recommends that the tentative revisions issued in 1959,[3] of the Standard Methods of Test for Conditioning Plastics and Electrical Insulating Materials for Testing (**D 618 – 58**)[2] be approved for reference to letter ballot of the Society for adoption as standard.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee, jointly with Committee D-9 on Electrical Insulating Materials, recommends for immediate adoption revision as follows of the Standard Method of Conditioning Plastics and Electrical Insulating Materials for Testing (**D 618 – 58**),[2] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual

Meeting in order that the revision may be referred to letter ballot of the Society:

Section 3(d).—Add the following test temperatures and tolerances to the list of standard test temperatures in this paragraph:

Test Temperatures	Tolerance, plus or minus
225 C (437 F)..........	3.0 C (5.4 F)
275 C (527 F)..........	3.0 C (5.4 F)
325 C (617 F)..........	4.0 C (7.2 F)
350 C (662 F)..........	5.0 C (9.0 F)
400 C (752 F)	6.0 C (10.8 F)
450 C (842 F)..........	8.0 C (14.4 F)
500 C (932 F)..........	10.0 C (18.0 F)
600 C (1112 F).........	12.0 C (21.6 F)

WITHDRAWAL OF STANDARD

The committee recommends the withdrawal of the Standard Method of Estimating Blocking of Plastic Sheets (**D 884 – 48**).[2]

REAPPROVAL OF STANDARDS

The committee recommends that all other standards under the jurisdiction of the committee not otherwise mentioned are to be continued as standard.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation of all other tentatives under the jurisdiction of the committee not otherwise mentioned in view of the fact that work is currently in process on revision thereof, or, in a few cases, insufficient experience has been had to justify advancement to standard.

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following tentatives:

Tentative Method of Measuring Absorbed Gamma Radiation by Fricke Dosimetry (D 1671 – 59 T):[3]

Title.—Revise the title to read "Method of Test for Absorbed Gamma Radiation Dose in the Fricke Dosimeter."

Tentative Specification for Polyethylene Molding and Extrusion Materials (D 1248 – 60 T):[5]

Section 1.—Change Section 1 to read as follows:

1. This specification provides for the identification of polyethylenes in such a manner that manufacturer and purchaser can agree on the substantial similarity of different commercial lots or shipments. It is not the function of this specification to provide engineering data nor to guide the purchaser in the selection of a material for a specific end use.

This specification covers requirements for polyethylene molding and extrusion compounds. Ordinarily, the tests listed in Section 8 are sufficient to characterize a material under this specification and it is recommended that routine inspection be limited to such tests.

Section 6.—Add a new Section 6 entitled "Contractual Requirements" to read as follows, renumbering present Section 6 and subsequent sections accordingly:

6. All the following requirements require agreement between manufacturer and purchaser as noted:

(*a*) Antioxidant or other additive and proportions:

Section 3(*b*) Classes A and B,
Same plus color: Section 3(*b*) Class B, and
Same plus amount, kind, particle size and means and degree of dispersion of carbon black: Section 3(*b*) Class C.

(*b*) Contamination level: Section 4(*b*).
(*c*) Nominal Melt Index: Section 4(*c*).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[7]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Mechanical Properties (P. Z. Adelstein, chairman) completed revisions on the Test for Rockwell Hardness of Plastics (D 785), the Test for Stiffness of Nonrigid

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[7] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Plastics by Means of a Torsional Test (D 1043), and Test for Stiffness in Flexure (D 747). A new rod stripping method for blocking characteristics and a new coefficient of friction test were developed. A change has been made in the specimen tolerances of the Test for Tensile Properties of Plastics (D 638). A new tentative tensile impact test has been approved. The revision of the Test for Tensile Properties of Plastic Films (D 882) is under active study. A round-robin test program is being completed on a tongue tear test, and laboratory investigations are under way on high-speed tension testing, compression testing using specimen jigs, and torsion pendulum studies. The Elmendorf Tear Test (D 689) and the Izod Impact Test (D 256) are also under study.

Subcommittee II on Radiation Effects (D. J. Metz, chairman), a joint subcommittee with Committee D-9, during the past year has concentrated on radiation dosimetry. Under the Dosimetry Section, two new task groups have been established to deal with the problems of neutron energy and flux measurements and to study the problems involved in mixed reactor radiations (principally neutrons and gamma rays).

An effort is being made to stimulate consideration of the reevaluation of standard dielectric measurements of materials in a radiation field.

Subcommittee III on Thermal Properties (D. L. Engle, chairman).—A revision of the Method of Test for Vicat Softening Point (D 1525) relaxes the rate of temperature rise requirement, for consistency with the ISO procedure. The procedure section of the Method of Test for Deformation Under Load (D 621) was clarified by the addition of explanatory notes. A round-robin test on a general zero-strength-time (ZST) test failed to demonstrate broad applicability, and the work was dropped. A round-robin test and a subcommittee ballot were successfully completed on a proposed test for ignition properties of plastics (the Setchkin ignition method). Task groups are working to accomplish a major revision of Method D 1238, to study the use of notched specimens in Method D 746, to revise Specifications D 538 and Method D 635, and to substantiate the applicability of Method D 1692 to flexible as well as rigid foams (jointly with Committee D-11).

Section L on Ablation Testing of Plastics, not yet two years old, enjoys an active membership of over one hundred. Task groups are active on flame testing, plasma jet testing, and definitions.

Subcommittee IV on Optical Properties (J. H. Ross, Jr., chairman).—Because of the greatly increasing interest in describing the optical clarity property (see-through distinctiveness of detail) of plastic films, a Tentative Method of Test for Transparency of Plastic Sheeting (D 1746 - 60 T) was prepared. Participating laboratories have modified their instruments to conform to the method and are participating in a round-robin evaluation of the method.

A proposed tentative method of test for yellowness index of transparent and opaque plastics has been drafted, balloted by the section and subcommittee, and is now ready for Committee D-20 letter ballot. The applicability of the proposed method for translucent plastics is currently under investigation.

The Standard Method of Test for Diffusion of Light by Plastics (D 636), was withdrawn in view of the forthcoming recommended practices for goniophotometry of transmitting and reflecting objects and materials being proposed by Committee E-12 on Appearance.

Subcommittee V on Permanence Properties (P. R. Graham, chairman).—

Section A on Effect of Light has completed a Recommended Practice for Determining Light Dosage in Artificial Weathering Apparatus which has been approved by the committee. Final approval by the Society awaits availability of a supply of standard fading chips. This method was backed by considerable research effort and has had a great deal of interest both in this country and abroad.

Revisions of Method D 1203 and Recommended Practice D 794 are being balloted upon in the subcommittee. This work is being conducted by Section B on Effect of Heat.

A round-robin study has been initiated in Section E on Laboratory Weathering to provide preliminary data on the performance of the Xenon Arc artificial weathering apparatus. Because of this instrument's reported similarity to sunlight exposure, this work is of decided interest.

Section H on Outdoor Weathering is revising Recommended Practice D 1435 to include more accurate definitions, possible photographic standards, and the standard fading chip.

Section J on Corrosivity is currently evaluating the galvanic current test for determining the corrosivity of molded plastics.

Recommended practices for the determination of resistance of plastics to both fungi and bacteria were written during 1960 by Section M on Effect of Microorganisms. Because of a possible safety hazard involved in the bacterial method, this method has been withheld until more information is available. However, the fungi method was submitted to Committee D-20 letter ballot with the bacterial method attached as information and for comments only.

The work of Section N on Environmental Stress Cracking is just getting under way.

Subcommittee VII on Analytical Methods (J. N. Jessup, chairman) is developing physical and spectrographic test methods. Section A on Physical Methods has revised Method D 792 on specific gravity and Method D 1505 on the density gradient technique. This latter method is presently under further revision to include alternate column preparation methods. A method for particle size and sieve analysis of plastic materials is about ready for committee ballot.

Section G on Gas Permeability has been reactivated and is presently undertaking to revise Method D 1434 on Gas Transmission Rate of Plastic Sheeting.

Section H on Spectrographic Methods has two task groups developing infrared methods for the analysis of polyolefins and vinyl chloride plastics. Round-robin test data are being analyzed with preliminary indications showing promise for the analysis of both materials.

Plans are being made to organize a group to investigate gas chromatography methods for plastics.

Subcommittee IX on Molds and Molding (K. A. Weston, chairman) has revised Recommended Practice D 796 and completed the new Recommended Practice for Injection Molding of Specimens of Polystyrene Molding and Extrusion Materials referred to earlier in this report. The procedures established permit the use of commercial injection molding machines for preparing these test specimens. Plans are being made to prepare a similar practice for styrene-butadiene plastic materials. Also completed is a recommended practice for transfer molding thermosetting materials and suggested mold designs.

Work is still in the formative stage for establishing methods for molding micro tension test specimens on miniature molding machines and a round-robin test will be conducted to evaluate the reliability of data obtained from such specimens. Establishing practices for preparing extruded test specimens is progressing and a round robin test will be organized to collect data for study.

Subcommittee X on Definitions (P. E. Willard, chairman) continued the preparation of definitions and abbreviations for terms relating to plastics. During the year, 29 new definitions were approved for addition to Definitions D 883. Liaison with Committee D-11 on Rubber and Rubber-Like Materials continued to resolve differences in definitions for rubber and plastics and thus to help in clearly establishing the areas of interest of each committee. All plastics definitions are to be combined in one document (D 883) and it is expected that this combination will be effected soon.

Subcommittee XI, on Editorial Review (G. M. Kline, chairman) has reviewed all proposed new and revised specifications, methods of test, and recommended practices, and submitted recommendations for editorial revision.

Subcommittee XII on Polyolefin Plastics (J. B. Howard, chairman) modified the specification covering polyethylene molding and extrusion materials (D 1248) to include the addition of stress-crack-resistant grade (IC5) to Table II. Also under active consideration were the possibility of changing the format of this specification to that of Specification D 1755 on poly (vinyl chloride) resins, and a revision of tension testing speeds. Action in the latter has been tabled.

Task groups made considerable progress on a draft of a proposed specification for polypropylene molding and extrusion compounds, and on a new method of preparing test sheets of polyethylene, designed to improve the precision of test results on crystallinity-dependent properties. Another task group is investigating the environmental stress-rupture test developed by Lander (Union Carbide Plastics Co.) as a possible improvement over Method D 1693.

Arrangements have been completed for improved liaison with Subcommittee V on Insulated Wire and Cable of Committee D-11, and with Subcommittees V and X of Committee D-20 through delegation of responsibilities for these functions as follows: Subcommittee V (Committee D-11): J. M. Campbell; Subcommittee V (Committee D-20): R. N. Carey; and Subcommittee X (Committee D-20): T. H. Meltzer.

The subcommittee accepted with regret the resignation of D. L. Engle as chairman of Section C; and is fortunate in obtaining R. H. Carey as the new chairman.

Subcommittee XIII on Statistical Techniques (R. H. Supnik, chairman) completed the proposed Tentative Reccommended Practice on Sampling appended to this report; and a recommended practice on conducting interlaboratory studies has been rewritten to conform to the desires of the subcommittee.

To implement systematic reporting of data in Committee D-20 methods, a task group has composed recommended practices regarding what values to report and when to discard data and to re-test.

Three indoctrination lectures were held as a continuing effort to provide Committee D-20 members with an understanding of statistical considerations and concepts as they may be applied to writing test methods and specifications. The speakers and their subjects were as follows: (1) Ross Johnson, The Martin Co., Md., "Effects of Parts Tolerances on Tolerances of the Finished Assembly"; (2) Hale Sweeney, Research Triangle Institute, N. C., "Nested Design in Round-Robin Studies"; and (3) T. W. Lashof, National Bureau of Standards, "Recommended Practice on Conducting Interlaboratory Tests on Paper."

Subcommittee XIV on Conditioning (G. M. Armstrong, chairman) has completed a revision of Methods D 618, as noted earlier in this report, in which additional standard test temperatures, higher than those now recommended, are proposed for conditioning or testing higher temperature-resistant plastics and insulating materials. The work of this subcommittee is a joint activity with Committee D-9.

Subcommittee XV on Thermoplastic Materials (W. D. Harris, chairman) established two new sections during the past year: Section Q on Polycarbonate Plastics which will develop test methods and specifications for this new and growing class of plastics; and Section D on Blown Thermoplastic Containers which will develop methods of characterizing such products of the rapidly growing blow molding industry.

The subcommittee has developed a specification for poly(vinyl chloride) resins which copes with the complex problem of a broad spectrum of materials by taking a new approach to specification writing based on a classification system which allows each material to be identified according to its particular combination of properties.

A specification for styrene-butadiene molding and extrusion materials was developed. The two viscosity methods for plastisols and organosols, based on the Brookfield instrument for low shear conditions and the Castor-Severs technique for high shear conditions have been accepted by the Society as noted earlier in this report. Specifications D 706, D 707, D 787, and D 789 have been revised, as noted earlier in this report, to bring them up to date.

Work under way includes the development of a specification for poly(vinyl chloride-vinyl acetate) resins; revision of Specification D 788 to refine deflection temperature under load testing and limits; a specification for rigid poly(vinyl chloride-vinyl acetate) compounds; a recommended practice for oven heat stability of poly(vinyl chloride) compositions; further revision of Specification D 789 to refine moisture conditioning and control procedures; investigation of methods for measuring plasticizer migration and volatilization; revision of Specifications and Methods D 1430 and D 1457 on halocarbon plastics; investigation of methods for preparing and testing vinyl dispersions and their fusion products; development of test methods for evaluating the environmental stress-cracking tendencies of blown thermoplastic containers; and the investigation of test methods for characterizing polycarbonate resins.

Subcommittee XVI on Thermosetting Materials (J. E. Hanna, chairman) prepared a new Tentative Specification for Epoxy Resins (D 1763 - 60 T) and a complete revision of Specification D 704 for melamine formaldehyde molding compounds. A revision of Specification D 700, Phenolic Molding Compounds, covering the addition of two new types has been approved for committee ballot. Considerable work in the field of epoxy resins and epoxy compounds is under investigation by Section E.

Subcommittee XVII on Thermoplastic Pipe and Fittings (Frank W. Reinhart, chairman) is a joint activity of The Society of the Plastics Industry and ASTM Committee D-20. Plans were developed to integrate more closely the work on standards of the SPI Thermoplastic Pipe Division and of Subcommittee XVII; these plans are being implemented. Section K on Nonpressurized Pipe was organized. Five tentative task groups were established in Section K on PVC, PE, ABS, and styrene-rubber drain and sewer pipe, and on electrical conduit.

Specifications for PE pipe have passed subcommittee letter ballot. Revisions of specifications for PVC pipe (D 1785) have passed subcommittee letter ballot. A test method for acetic acid immersion of ABS pipe and fittings has passed subcommittee letter ballot. Test methods for acetone immersion of PVC pipe and fittings for dimensions, and a revision of D 1599 are being submitted to subcommittee letter ballot. Work is being initiated on specifications for conduit. Specifications for ABS pipe, PVC fittings, ABS fittings, styrene-rubber sewer and drain pipe, PVC drain pipe, and cements for ABS pipe and fittings are being submitted to subcommittee letter ballot.

Test methods are being developed for chemical resistance, impact resistance, measurement of expansion, measurement of dimensions, detection of weeping, and surge pressure strength. Revisions of the test methods for determining short-term and long-term strengths (D 1598 and D 1599) are being prepared. The method for determining long-term strengths and working pressures of thermoplastic pipe developed in the Thermoplastic Pipe Division of The Society of the Plastics Industry is being reviewed; a round-robin testing program on this proposal was established. Formulas for relating circumferential stress, dimensions, and internal pressure are also under consideration with a view to adopting one as a standard for use in the work of the subcommittee.

The request that the USA be represented in ISO/TC5/SC6 on Plastic Pipe has been granted. A USA National Committee to handle the work for the American Standards Assn. with representation from ASTM Committee D-20, The Society of the Plastics Industry, the Society of the Plastics Engineers, Manufacturing Chemists Assn., and Section Committees B16, B31, and B72 in the American Standards Assn. has been proposed to ASA.

Subcommittee XVIII on Reinforced Plastics (H. A. Perry, chairman).—Section A has continued activity on flat sheets in cooperation with Committee D-9. Section B is continuing to develop test methods, dimensional standards, and specifications on reinforced plastics pipe in cooperation with The Society of the Plastics Industry. Section C has been closely coordinated with the Commercial Standards groups and has recommended a specification on corrugated sheets for committee ballot. Section D was reactivated to establish the requirements for test methods and specifications on laminating resins, to review existing test methods which have been standardized by ASTM that may meet the requirements, and to determine the need for further work in this area. Section E has continued the development of a specification on glass fabrics, heat-cleaned and finished for plastics laminates, and has undertaken new work in the area of yarns and rovings, particularly for filament winding. Section H was formed to undertake the development of methods of sample preparation and test methods, specific to reinforced plastics. Section I was formed to develop methods of inspection and non-destructive testing specific to reinforced plastics. Section J was formed to develop test methods and specifications on prepreg reinforcements and premix molding compounds.

Subcommittee XIX on Plastic Film and Sheeting (K. A. Kaufmann, chairman).—Section A prepared the new Method of Test for Evaluation of Welding Performance for Poly(Vinyl Chloride) Structures (D 1789 - 60 T). Work on a proposed tentative specification for rigid poly(vinyl chloride) sheet is complete and has been submitted for committee approval. Currently the section is working on a similar tentative specification for poly(vinyl chloride) copolymer sheet.

Section B completed work on a Proposed Method of Test for the Kinetic

Coefficient of Friction of Polyethylene Film. Currently under subcommittee ballot is a proposed Tentative Specification for Polyethylene Film; and round-robin studies are in progress on a proposed method of test for determining the impact resistance of thick polyethylene film.

Section D made further revisions in the Specification for Cast Methacrylate Plastic Sheets, Rods, Tubes and Shapes (D 702 - 60 T). Further work on measurement of optical properties of rods, tubes, and shapes is in progress.

Section F is studying test methods for degree of surface treatment of polyethylene film through round-robin activity. The subcommittee is presently considering revision of the Specification for Biaxially Oriented Styrene Plastic Sheet (D 1463) to bring it more in line with current commercial products.

Subcommittee XX on Cellular Plastics (S. Steingiser, chairman) continues its development of methods of test for water absorption, water vapor transmission, permanence properties, flexure and dimensional stability.

Liaison has been established with Committee C-16 on Thermal Insulating Materials for development of test methods on thermal conductivity. The heat flow meter method is now being processed through Committee C-16 while the line probe method is being redrafted in Subcommittee XX for either publication as information or promulgation as a tentative.

Work on the open and closed cell method continues with some effort to develop a new method of test. However, no definitive action can yet be taken without further laboratory investigation. The original method written some years ago will still need to be held until such work is done.

Specification work in polystyrene form is still in its final stages and it is hoped that difficulties there are resolved shortly. Specifications on rigid urethane foam are in process now as data are being developed.

Subcommittee XXI, U. S. A. National Committee, ISO/TC 61 (A. Webber, chairman).—A practical basis for simplifying and increasing international plastics trade was another important step toward maturity as the International Standards Organization's Technical Committee on Plastics (ISO/TC 61) completed its tenth annual week-long meeting in Prague, Czechoslovakia. With keener competition to American industry, particularly from Europe's highly successful common markets, the inner six and outer seven, ISO/TC 61 standards become the working tools for competing on a par with other nations. Producers and consumers alike find internationally-agreed methods, specifications, and terminology useful, both in international trade and in their own national standards work. In addition, the exchange of purely technical information, apart from commercial work, is smoother, simpler, and less expensive.

It was with these ideas in mind that 113 delegates from 15 nations approved for ballot three more methods of test for plastics: (1) torsion stiffness as a function of temperature; (2) maximum temperature and rate of rise during setting of unsaturated polyesters; and (3) tensile stress-strain properties. These bring to 36 the total of draft ISO Recommendations sanctioned by TC 61 for final preparation as international methods. New methods and methods for re-balloting of the 36 member nations of TC 61 included: (1) resistance to color changes by sunlight, (2) chemical resistance, (3) resistance to light from the carbon-arc, (4) analysis of monomer and low molecular weight polymer in polyamides, (5) viscosity of polyamides in concentrated solution, and (6) water content of polyamides. Five ISO recommendations prepared by the Technical

Committee were published during the past year which bring to nine the total of published ISO recommendations for plastics.

Thus, the ISO Technical Committee on Plastics has retained its front-running position as the most productive of the ninety-seven ISO committees dealing with industrial products and processes.

Subcommittee XXII on Plastics for Tooling (J. J. Mele, chairman) has decided upon the scope of each of its five sections: Section A on Ingredient Specifications; Section B on Plastics Specifications; Section C on Ingredient Test Methods; Section D on Plastics Test Methods; and Section E on Nomenclature. It has been agreed that sections working on ingredient specifications and test methods will include in their scope all resin and curing agent combinations up to the point when the system is considered set. From this point on, sections working on plastics specifications and test methods will take over.

Subcommittee letter ballots have been distributed on a plastic test method on shrinkage. The Epoxy Resin Formulators and the Plastic Tooling Section of The Society for the Plastic Industry are also compiling data and preparing test methods and specifications. Wherever possible, Subcommittee XXII would first consider incorporating work and results available from these two organizations and with their respective permission and assistance, prepare letter ballots according to ASTM procedures.

This report has been submitted to letter ballot of the committee, which consists of 263 voting members; 160 members returned their ballots, of whom 147 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. B. DECOSTE,
Chairman.

C. F. FERRARO,
General Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-20 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method of Test for:

Particle Size (Sieve Analysis) of Plastic Materials (D 1921 - 61 T),
Propagation Tear Resistance of Plastic Film and Thin Sheeting (D 1922 - 61 T), and
Tensile Properties of Thin Plastic Sheet by Means of Incline Plane Test (D 1923 - 61 T).

New Tentative Recommended Practice for:

Determining Resistance of Plastics to Fungi (D 1924 - 61 T).

Revision of Tentative Method of Test for:

Coefficients of Friction of Plastic Film (D 1894 - 61 T),
Tensile Properties of Plastics (D 638 - 60 T), and
Tensile Properties of Thin Plastic Sheeting (D 882 - 56 T).

Revision of Tentative Definitions of Terms Relating to:

Plastics (D 883 - 61 T).

Revision of Tentative Abbreviations of Terms Relating to:

Plastics (D 1600 - 60 T).

Consolidation of:

Tentative Nomenclature of Descriptive Terms Pertaining to Plastics (D 675 - 60 T) and Tentative Definitions of Terms Relating to Plastics (D 883 - 61 T), the title and designation of the combined documents to be "Tentative Nomenclature of Terms Relating to Plastics (D 883 - 61 T)."

These recommendations were accepted by the Standards Committee on September 12, 1961, and the new and revised tentatives appear in the 1961 Book of ASTM Standards, Part 9.

The following additional recommendations of Committee D-20 were accepted by the Standards Committee on the dates indicated; the revision of Tentative Definitions D 883 appear in Part 9 of the 1961 Book of Standards, and the others are available as separate reprints:

New Tentative Method of Test for:

Yellowness Index of Transparent and Opaque Plastics (D 1925 - 62 T) (Accepted Jan. 3, 1962).

Revision of Tentative Definitions of Terms Relating to:

Plastics (D 883 - 61 T) (Accepted Jan. 3, 1962).

Revision and Reversion to Tentative of Standard Method of Test for:

Volatile Loss from Plastic Materials (D 1203 - 55) (Accepted Dec. 11, 1961).

Revision and Reversion to Tentative of Recommended Practice for:

Determining Permanent Effect of Heat on Plastics (D 794 - 49) (Accepted Jan. 12, 1962).

REPORT OF COMMITTEE D-21
ON
WAX POLISHES AND RELATED MATERIALS

Committee D-21 on Wax Polishes and Related Materials held one meeting during the year: in Cincinnati, Ohio, Feb. 2 and 3, 1961. In addition, the subcommittees met in Chicago, Ill., May 18, 1961.

Additions and changes during the year brought the membership of the committee to 70, of whom 32 are classified as producers, 12 as consumers, and 26 as general interest members, with one consulting member.

TENTATIVES CONTINUED WITHOUT REVISION

The committee reviewed all of the tentatives under its jurisdiction and recommends their continuation without revision.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature (J. V. Steinle, chairman) is continuing its work on definitions for inclusion in a glossary of terms relating to wax polishes. Several new definitions are currently under letter ballot to the subcommittee.

Subcommittee II on Raw Materials (Herbert Mellan, chairman) is reviewing methods of determining the melting point of waxes with the aim of establishing a standard method for wax polish raw materials. A compilation of proposed test methods for emulsion polymers used in wax polishes is also in preparation. In addition, work is continuing on the development of a method for the determination of resin content of natural waxes.

Subcommittee III on Chemical and Physical Testing (J. G. Sinsheimer, chairman) is developing a test method for determining the freezing-thawing stability of emulsion polishes. Other task groups are currently working on methods for the determination of total solids and silicone content of solvent-based polishes.

Subcommittee IV on Performance Tests (C. S. Kimball, chairman) is completing a revision of the proposed method of test for static coefficient of friction of waxed surfaces, which was published in 1954 as information only. The revised method will be submitted to letter ballot of the committee for approval as a tentative. A method for determining the adhesion or "powdering" of floor polishes is also nearing completion. In addition, task groups are investigating methods of determining discoloration and recoatability of emulsion polishes.

Subcommittee V on Specifications (William Joy, chairman) is continuing the preparation of a specification for industrial-type water emulsion floor waxes.

This report has been submitted to letter ballot of the committee, which consists of 70 members; 45 members returned their ballots, of whom 39 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. W. WALTON,
Chairman.

BAYARD S. JOHNSON,
Secretary.

REPORT OF COMMITTEE D-22

ON

METHODS OF ATMOSPHERIC SAMPLING AND ANALYSIS*

Committee D-22 on Methods of Atmospheric Sampling and Analysis met on Jan. 31 and Feb. 1, 1961, in Cincinnati, Ohio.

The committee membership at the time of the January–February, 1961, meeting totaled 100.

The committee was represented by Dr. M. Katz at a meeting of an ad hoc committee held in New York City, December 6, 1960, to consider a procedure for the development of a jointly-sponsored manual on recommended methods for the sampling and analysis of air.

The ad hoc group had proposed that the work be done by an intersociety committee under the administrative cognizance of the American Public Health Assn. In considering this proposal, Committee D-22 had some reservations about the feasibility of the project as outlined, but it did agree in principle that the manual was much needed, and the committee was prepared to cooperate fully in the work.

NEW TENTATIVE

The committee recommends for publication as tentative the Method of Test for Mass Concentration of Particulate Matter in the Atmosphere (Continuous-Measurement Light-Scattering Method) as appended hereto.[1]

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Method of Test for Particulate Matter in the Atmosphere (Optical Density of Filtered Deposit) (**D 1704 – 59 T**)[2] be approved for reference to letter ballot of the Society for adoption as standard without revision.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Units (E. R. Hendrickson, chairman) has prepared proposed Recommended Tables of Conversion Units and Factors.

Subcommittee II on Sampling and Analysis (H. Chapman, chairman).—Task Group C on Sampling for Particulate Matter is preparing a guide showing what materials can be collected by filtration. Task Group D on Particle Size is preparing a procedure for the microscopic examination of samples collected on a millipore filter, and another procedure on the preparation of millipore samples for electron microscopy. Task

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] The new tentative appears in the 1961 Book of ASTM Standards, Part 10.

[2] 1960 Supplement to Book of ASTM Standards, Part 10.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Group E on Stack Sampling is developing a standard procedure suitable to most field problems. Task Group F on Miscellaneous Methods is being reorganized into two sections, one on corrosion and another on vegetation sampling. Task Group G on Lead Peroxide (SO_2) Candle is planning field trials to ascertain the most suitable method of preparation and handling of candles. Other Task Groups are developing methods for determining SO_2, SO_3, organic lead, formaldehyde, acrolein, and polynuclear hydrocarbons.

Subcommittee III on Instrumentation (M. D. Thomas, chairman) completed the proposed Tentative Method of Test for Mass Concentration of Particulate Matter in the Atmosphere (Light-Scattering Method). Procedures for continuous analysis of oxidant and oxides of nitrogen are near completion. Procedures are being considered for directional dustfall sampling, fluoride recorders, and sulfuric acid analysis.

This report has been submitted to letter ballot of the committee, which consists of 66 voting members; 35 members returned ballots, of whom 31 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

J. CHOLAK,
Chairman.

A. T. ROSSANO, JR.,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-22 presented to the Society through the Administrative Committee on Standards the recommendation that the Recommended Practice for Conversion Units and Factors Relating to Atmospheric Analysis be published as tentative. This recommendation was accepted by the Standards Committee on September 12, 1961, and the new tentative recommended practice appears in the 1961 Book of ASTM Standards, Part 10, bearing the designation D 1914 – 61 T.

REPORT OF COMMITTEE D-23
ON
CELLULOSE AND CELLULOSE DERIVATIVES*

Committee D-23 on Cellulose and Cellulose Derivatives held one meeting during the year: in New York City on September 14, 1960. The Executive Subcommittee also met in New York City on February 23, 1960.

The committee consists of 47 members, of whom 14 are classified as producers, 11 as consumers, and 22 as general interest members.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1960 Annual Meeting, Committee D-23 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method of Test for:

Pentosans in Cellulose (D 1787 – 60 T).

Withdrawal of Tentative Method of Test for:

Pentosans in Cellulose (Aniline Acetate Colorimetric Method) (D 1438 – 56 T).

These recommendations were accepted by the Standards Committee on September 29, 1960, and the new Tentative Method D 1787 appears in the 1960 Supplement to Book of ASTM Standards, Part 6.

PROPOSED METHOD TO BE PUBLISHED AS INFORMATION

The committee recommends for publication as information only the Proposed Methods of Test for Carboxyl Content of Cellulose as appended hereto.[1]

REVISION OF TENTATIVE

The committee recommends revisions as appended hereto[2] of the Tentative Methods of Testing Sodium Carboxymethylcellulose (**D 1439 – 58 T**)[3] and continuation of the method as tentative.

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends revisions as appended hereto[2] of the Standard Methods of Testing Cellulose Acetate (**D 871 – 56**)[3] and reversion of the method to tentative.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods of Test for:

Moisture in Cellulose (**D 1348 – 58 T**),[3] and

Solubility of Cellulose in Sodium Hydroxide (**D 1696 – 59 T**).[4]

EDITORIAL CHANGES

The committee recommends editorial changes as follows in the Tentative

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] Subsequent to the Annual Meeting, this proposed method was accepted for publication as tentative; see editorial note, p. 593.

[2] The revised method appears in the 1961 Book of ASTM Standards, Part 6.

[3] 1958 Book of ASTM Standards, Part 6.

[4] 1959 Supplement to Book of ASTM Standards, Part 6.

Method of Test for Intrinsic Viscosity of Cellulose (**D 1795 - 60 T**):[5]

Section 10(b).—Change the last sentence to read: "Repeat at least twice and average the observations, which should not differ by more than 0.3 per cent."

Section 11.—Add a new Paragraph (*e*) to read as follows:

> (*e*) "Based on an interlaboratory test, the reproducibility among laboratories should be ±3 per cent."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[6]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Definitions (Kyle Ward, Jr., chairman) is developing revisions of the Tentative Definitions of Terms Relating to Cellulose and Cellulose Derivatives (D 1695 - 60 T).

Subcommittee II on Cellulose (E. S. McColley, chairman) has prepared the Proposed Methods of Test for Carboxyl Content of Cellulose, and has also recommended the editorial revisions of Method D 1795 as noted earlier in this report.

A method for measurement of whiteness by calculating the values from a G.E. Recording Spectrophotometer reflectance curve is under consideration. A solution color method based on transmission of a cadmium complex (Cadoxene) solution at 540 mμ is also being considered.

The Task Group on Ash and Ash Constituents is studying both an ash procedure similar to TAPPI Method T 211m-58 and a sulfated ash procedure published in Sweden.

The Task Group on Chromatographic Methods has analyzed a series of ICCA pulps and mixtures of pure sugars, with and without simulated hydrolysis, and prepared a proposed procedure.

Subcommittee III on Organic Esters (L. B. Genung, chairman) has completed the revision of Method D 871 to include new test procedures for acetyl (alternate), hydroxyl, primary hydroxyl, intrinsic viscosity, color, combined sulfur or sulfate, and heat stability as appended to this report. A similar revision of Method D 817 is in progress. All the above listed new test procedures have been added, and the method is extended to include cellulose acetate propionates as well as acetate butyrates.

Subcommittee IV on Inorganic Esters (C. B. Gilbert, chairman) is in the process of revising Sections 16 and 17 of Method D 301 to conform to the single dilution tests described in Method D 1720 - 60 T, under the jurisdiction of Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

Subcommittee V on Cellulose Ethers (R. W. Swinehart, chairman).—The Task Group on Purified Sodium Carboxymethyl cellulose has prepared the alternate method for degree of etherification and the colorimetric procedure for sodium glycolate as appended to this report as a revision of Method D 1439.

This report has been submitted to letter ballot of the committee, which consists of 47 voting members; 29 have returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

F. A. SIMMONDS,
Chairman.

W. W. BECKER,
Secretary.

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[5] 1960 Supplement to Book of ASTM Standards, Part 6.

[6] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-23 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method of Test for:

Chromatographic Analysis of Chemically Refined Cellulose (D 1915 - 61 T), and
Carboxyl Content of Cellulose (D 1926 - 61 T).

These recommendations were accepted by the Standards Committee on September 12 1961, and the new tentative methods appear in the 1961 Book of ASTM Standards Part 6.

REPORT OF COMMITTEE D-24
ON
CARBON BLACK*

Committee D-24 on Carbon Black and its subcommittees held two meetings during the year: in Atlantic City, N. J. on June 28, 1960, and in Cincinnati, Ohio, on January 31, 1961.

New Tentative

The committee recommends for publication as tentative the Method for Sampling Bulk Shipments of Carbon Black, as appended hereto.[1]

Revision of Tentative

The committee jointly with Committee D-11 on Rubber and Rubber-Like Materials, recommends revision as follows of the Tentative Specification for Carbon Blacks Used in Rubber Products **(D 1765 – 60 T)**[2] and its continuation as tentative:

Table II.—Revise modulus limits for GPF black to read as follows:

Type and Minutes at 293 F	Stress at 300 per cent Elongation, psi	
	Min	Max
GPF		
15	−400	+100
30	−525	−25

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

Activities of Subcommittees

Subcommittee I on Physical Tests (J. F. Svetlik, chairman) is continuing work on a test method for mass strength of pelleted carbon black.

Subcommittee III on Optical Tests (J. E. Smith, chairman) has reviewed the benzene discoloration method (D 1618) and recommends continuation as tentative without revision.

Subcommittee IV on Nomenclature (J. F. Gifford, chairman) is considering methods of classifying rubber grade carbon black and possible nomenclature for new grades.

Subcommittee V on Sampling (R. O. Treat, chairman) prepared the Tentative Method for Sampling Bulk Shipments of Carbon Black appended to this report.[1]

Subcommittee VII on Specifications (J. F. Gifford, chairman) is working in conjunction with Subcommittee XXIX of Committee D-11 on Rubber and Rubber-Like Materials on carbon blacks in styrene-butadiene rubbers (SBR). The two groups prepared the revisions of Tentative Specifications for Carbon Blacks Used in Rubber Products (D 1765) referred to earlier in the report.

This report has been submitted to letter ballot of the committee, which consists of 56 members; 41 members returned their ballots, of whom 32 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

N. P. Bekema,
Chairman.

T. D. Bolt,
Secretary.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] The new tentative appears in the 1961 Book of ASTM Standards, Part 9.

[2] 1960 Supplement to Book of ASTM Standards, Part 9.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE D-25
ON
CASEIN AND SIMILAR PROTEIN MATERIALS

Committee D-25 on Casein and Similar Protein Materials held two meetings during the year: on May 26, 1960, in Chicago, Ill., and on Feb. 24, 1961, in New York, N. Y.

The committee consists of 25 members, of whom 7 are classified as producers, 8 as consumers, and 10 as general interest members.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I, Editorial and Nomenclature (M. E. Stonebreaker, chairman) has prepared a series of proposed definitions of terms which are being submitted to the full committee for an expression of approval.

Subcommittee III on Chemical Tests (E. T. McBroom, chairman) is continuing the development of methods for fat in casein and isolated soy protein and a procedure to determine the bacterial count in these materials.

Subcommittee IV on Physical Tests (E. Maynard, chairman) has completed a 3-year cooperative study on determining the Brookfield viscosity, in apparent centipoises, on a 15 per cent solid casein solution at 25 C (77 F). Variables studied were type of mixer, speed (rpm) of spindle, type of spindle, and pH. These data show that a satisfactory standard method should include a specified mixer, speed of spindle specified to ±10 per cent, and that a drop of defoamer be added to the casein solution.

Twelve laboratories cooperatively developed data for a method for wax pick and I.G.T. pick strength determinations on samples of soy protein coated paper. The method showed good reproducibility and will be submitted to subcommittee letter ballot.

Work is continuing on sieve analysis and tests for foaming foreign matter, insolubles, minimum alkali, and odor.

Respectfully submitted on behalf of the committee,

L. E. CLARK, JR.,
Chairman.

J. R. BORST,
Secretary.

REPORT OF COMMITTEE D-26

ON

HALOGENATED ORGANIC SOLVENTS*

Committee D-26 on Halogenated Organic Solvents held two meetings during the year: in Atlantic City, N. J., on June 30 and July 1, 1960, and in Cincinnati, Ohio, on Feb. 2 and 3, 1961. Subcommittee IV held a special meeting in New York City in October, 1960; and Subcommittee III met in Pittsburgh in March, 1961.

The committee consists of 67 members, of whom 21 are classified as producers, 31 as consumers, and 15 as general interest members.

NEW TENTATIVE

The committee recommends for publication as tentative the proposed Tentative Method of Test for Relative Evaporation Time of Halogenated Hydrocarbons and Their Admixtures as appended hereto.[1]

The recommendation in this report has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Vapor Degreasing (V. B. Smesrude, chairman) is preparing a monograph on all aspects of this process.

Subcommittee III on Cold Cleaning (M. Z. Poliakoff, chairman) is working on a list of halogenated hydrocarbons as requested in a survey made of major consumers. The subcommittee is also studying the problems of promoting the safe use of cold cleaners and of expressing the hazard of these solvents considering both toxicity and flammability.

Subcommittee IV on Test Methods (R. T. K'Burg, chairman) prepared the new method appended to this report. The subcommittee is studying ASTM procedure for determination of boiling range, specific gravity, stability, and acid acceptance of trichloroethylene and perchloroethylene; and also determination of explosibility of volatile liquid solvent mixtures. Work is under way on boiling range, specific gravity, flash points, moisture, acidity or alkalinity, residue, and color of solvent mixtures.

Subcommittee V on Industrial Hygiene (N. E. Whitman, chairman) is submitting definitions of dermatitis, toxicity, etc., and editing all reports from the viewpoint of the Industrial Hygienist.

Subcommittee VI on Dry Cleaning (A. P. Stetser, chairman) will be formally organized in June, 1961, at the Annual Meeting.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] The new tentative appears in the 1961 Book of ASTM Standards, Part 10.

[2] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.

This report has been submitted to letter ballot of the committee, which consists of 67 members; 32 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. D. McMASTER,
Chairman.

W. L. CAMPBELL,
Secretary.

REPORT OF COMMITTEE D-27
ON
ELECTRICAL INSULATING LIQUIDS AND GASES*

Committee D-27 on Electrical Insulating Liquids and Gases held three meetings during the year: in Atlantic City, N. J., during the Annual Meeting of the Society, on June 29 to July 1, 1960; in Williamsburg, Va., on Nov. 1–4, 1960; and in Washington, D.C., on March 20–22, 1961.

At the time of the Washington meeting the membership total was 105, of whom 78 were voting representatives; 28 were classified as producers, 58 as consumers, and 19 as general interest members.

The following liaison representatives have been appointed during the year:

E. R. Thomas as representative of Committee D-27 on Technical Committee 10 on Insulating Oils of the International Electrotechnical Commission.

R. G. Call as representative of Committee D-27 on the Power Station Chemistry Group of the Edison Electric Inst.

E. L. Raab as representative on the Transformer Committee on Askarels of AIEE.

Paul Narbut as representative on Section VI of the Committee on Gaseous Dielectrics of AIEE.

J. C. Parker as representative on the Insulated Conductors Committee of AIEE.

The committee lost two of its members of long standing in the deaths of Jayson C. Balsbaugh on July 17, 1960, and O. Emmett Fawcett on August 7, 1960.

Frank M. Clark, who retired from active service with the General Electric Co. on July 1, 1960, has been elected an honorary member of the committee.

E. R. Thomas will attend the meeting of Committee 10 on Insulating Oils of the International Electrotechnical Commission (IEC) which will be held on June 28–30, 1961 in Interlaken.

One item for discussion at this meeting is international standardization of a sludge test which is somewhat similar to the ASTM sludge accumulation test (D 1314). The fact that Committee D-27 has now approved a test method for dielectric strength using the VDE gap and electrodes (D 1816) will be of interest to the IEC committee since it is favorably disposed toward this type of apparatus.

Committee 1 of the International Conference on Large Electric Systems (CIGRE) has agreed on the design of a cell for power factor determinations which is quite similar to the ASTM cell and should give equivalent results.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1960 Annual Meeting, Committee D-27 presented to the Society through the Administrative Committee on Standards the following recommendations which were accepted on the dates indicated:

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

New Tentative Methods of Test for:

- Dielectric Strength of Insulating Oil of Petroleum Origin (Using VDE Electrodes) (D 1816 – 60 T) (Accepted Sept. 29, 1960),
- Hydrolyzable Chlorine Compounds in Chlorinated Aromatic Hydrocarbons (Askarels) (D 1820 – 60 T) (Accepted Dec. 29, 1960),
- Inorganic Chlorides in Askarels (D 1821 – 60 T) (Accepted Dec. 29, 1960), and
- Gas Content (Non-Acidic) of Insulating Liquids by Displacement with Carbon Dioxide (D 1827 – 61 T) (Accepted Feb. 15, 1961).

New Tentative Continuity Specifications for:

- Electrical Insulating Oil for Low Pressure Cable Systems (D 1818 – 60 T) (Accepted Dec. 29, 1960), and
- Electrical Insulating Oil for High Pressure Cable Systems (D 1819 – 60 T) (Accepted Dec. 29, 1960).

The new Tentative Method D 1816 appears in the 1960 Supplement to Book of ASTM Standards, Part 9. The other new methods and specifications are available as separate reprints.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto:[1]

Tentative Methods of Test for:

- Approximate Acidity and Polar Contamination in Used Mineral Transformer Oil by Spot Tests,
- Coefficient of Thermal Expansion of Electrical Insulating Liquids of Petroleum Origin, and Askarels, and
- Oxidation Characteristics of Mineral Transformer Oils.

REVISION OF TENTATIVE

The committee recommends revisions as appended hereto[1] of the Tentative Method of Test for Approximate Acidity of Used Electrical Insulating Oil of Petroleum Origin (**D 1534 – 58 T**)[2] and continuation of the method as tentative.

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends revisions as appended hereto[1] of the Standard Method of Test for 2,6-Ditertiary-Butyl Para-Cresol in New Electrical Insulating Oils (**D 1473 – 60**)[2] and reversion of the method to tentative.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as follows of the Standard Method of Test for Water in Insulating Liquids (Karl Fischer Method) (**D 1533 – 60**)[3] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Section 4.—Add the following Note to Paragraph (*c*), renumbering the present Note 2 and subsequent notes accordingly:

> *Note 2.*—In a sealed system, recleaning and redrying of the stirrer are not necessary for routine use.

In Paragraph (*d*) delete the words "30-ml capacity."

Section 6(*a*).—Add the following new Note 4 and renumber the subsequent notes accordingly:

> *Note 4.*—More dilute solutions of Karl Fischer reagent may be used on samples of low water content.

Section 7.—Add a subtitle to read "Method A—Methanol-Water Solution."

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[1] The new and revised tentatives appear in the 1961 Book of ASTM Standards, Part 11.

[2] 1958 Book of ASTM Standards, Part 9.

[3] 1960 Supplement to Book of ASTM Standards, Part 9.

New Section.—Add the following new Section 8, renumbering the present Section 8 and subsequent sections accordingly:

8. *Method B—Sodium Tartrate Dihydrate.*—(*a*) The water content of the sodium tartrate should be 15.66 ± 0.05 per cent. This value should be checked by drying a preweighed sample to a constant weight in an oven at 150 C.

(*b*) From a tared weighing bottle or by using a glass capsule, add 0.025 to 0.05 g of sodium tartrate, the weight of which is known to the nearest 0.001 g, to 100 ml of dried solvent as in Section 7(*a*). Titrate to the same end point with Karl Fischer reagent and record the amount of reagent.

(*c*) Calculate the water equivalent of the Karl Fischer reagent as follows:

$$F = \frac{\text{g sodium tartrate} \times 0.1566}{\text{ml Karl Fischer reagent}}$$

(*d*) Repeat the standardization and average the two values.

WITHDRAWAL OF TENTATIVE

The committee recommends the withdrawal of the Tentative Method of Test for Free Chlorides in Askarels (**D 1811 – 60 T**).[3] This recommendation is made since the Method of Test for Inorganic Chlorides in Askarels (D 1821 – 60 T) is a more complete method.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee A on Mineral Oils (E. L. Raab, chairman) has observed a developing trend to examine insulating oils for thermal decomposition in relatively closed systems from which oxygen is excluded. The use of infrared spectroscopy has increased in interest and a non-technical explanation is in preparation. The subcommittee has completed the preparation of continuity specifications for light and heavy cable oils referred to earlier in the report. The work of the subcommittee ties in closely with foreign developments in the IEC, and the action taken by that body and CIGRE on power factor, dielectric strength, and sludge testing is awaited with interest.

Subcommittee B on Synthetics (T. K. Sloat, chairman).—With the increasing number of methods of testing askarels, the committee is recommending their issuance as separate methods. Methods of Testing Askarels (D 901) then will become a reference to the methods and will contain only statements on significance and definitions of the various tests, similar to the arrangement of Methods for Testing Electrical Insulating Oils (D 117 – 58). As the result of work by the subcommittee, eight separate methods for testing askarels will be ready for publication this year.

Work on silicones and synthetics is proceeding slowly in this relatively new field.

Subcommittee C on Gases (R. B. Blodgett, chairman) has set up two groups, one on pure gases such as nitrogen, and a second group on halogenated gases. Considerable progress has been made in the development of a specification for nitrogen to meet the service requirements. Sulfur hexafluoride will also be considered.

Subcommittee O on Chemical Tests (H. W. McCulloch, Jr., chairman) prepared the following new methods and revisions which are included in the report: new tests for Approximate Acidity and Polar Contamination in Used Transformer Oil by Spot Tests, and Oxidation Characteristics of Mineral Transformer Oils, and revisions of Methods D 1534, D 1473, and D 1533.

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Progress has been reported in metal determination and tests for stability of askarels.

Subcommittee N on Electrical Tests (H. O. Bennett, chairman) prepared the new Method for Dielectric Strength using the German VDE electrodes (D 1816), referred to earlier. A revision of Method D 877 is under way to make apparatus and statistical sections correspond with those of the new Method D 1816. Round-robin tests are in progress on methods for evaluating the dielectric strength of insulating gases and the gassing tendency of insulating oils. Stability tests for combinations of oil and insulating paper are being considered in cooperation with Subcommittee VIII on Paper of Committee D-9 on Electrical Insulating Materials, while a test for the electric stability of cable oils is now ready for ballot approval. The dielectric strength of gases and apparatus for the same determination on small liquid samples are under consideration. A study is being made of a test to determine power factor changes occurring in insulating oil during its oxidation.

Subcommittee P on Physical Tests (J. C. Parker, chairman) prepared the new Method for Coefficient of Thermal Expansion referred to earlier. The use of carbon dioxide as a displacing medium has resulted in the new Method for Gas Content of Insulating Liquids (D 1827). The sampling procedures are being scrutinized, while methods for infrared absorption, interfacial tension, and emulsion are under consideration. Volatility or evaporation is being studied to determine some basis for a test procedure. The dew point of gases and vapor pressure measurements on halogenated gases are being examined.

This report has been submitted to letter ballot of the committee which consists of 105 members, of whom 78 are voting representatives; 47 members returned their ballots of whom 42 have voted affirmatively and 0 negatively

Respectfully submitted on behalf of the committee,

EARL R. THOMAS,
Chairman.

CHARLES A. JOHNSON,
Recording Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-27 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method of:

Test for Thermal Stability of Chlorinated Aromatic Hydrocarbons (Askarels) (D 1936 – 62 T),

Oxidative Aging of Electrical Insulating Petroleum Oils by Open-Beaker Method (D 1934 – 62 T), and

Test for Steam Emulsion of Electrical Insulating Oils of Petroleum Origin (D 1935 – 62 T).

New Tentative Specification for:

Nitrogen Gas as an Electrical Insulating Material (D 1933 – 62 T).

The new tentative methods and specification are available as separate reprints and they appear in the 1962 Compilation of ASTM Standards on Electrical Insulating Liquids and Gases. The date of approval by the Standards Committee is January 3, 1962.

REPORT OF ALCA-ASTM JOINT COMMITTEE
ON
LEATHER*

The ALCA-ASTM Committee on Leather (joint with the American Leather Chemists' Assn.) held two meetings during the year: On Dec. 8 and 9, 1960, in New York, N. Y., and on March 14 and 15, 1961, in Philadelphia, Pa.

NEW TENTATIVES

The committee recommends for publication as tentative the Method of Test for Cold-Crack Resistance of Upholstery Leather as appended hereto.[1]

This recommendation has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature, Definitions, and Applicability (R. M. Lollar, chairman) has continued to define and interpret precision and significance statements. In those methods where the report of results takes the form of ordinal or scaler values and the use of standard deviation as a measure of precision is inappropriate, the subcommittee is urging that the sections involved secure some numerical data on the reproducibility of these ordinal or scaler values. Emphasis should be placed on between-laboratory variability in the results, the skin to skin variability, and, where possible, the within skin variability. While this will not require elaborate statistical analysis, some statement along these lines will aid in interpretation of the results.

The subcommittee has reviewed drafts of four methods of test and was asked to develop definitions to cover the transfer of material from leather to foreign materials and from foreign materials to leather and to clarify the meaning of the words staining and bleeding.

Subcommittee II on Sampling and Conditioning for Physical Testing (C. W. Mann, chairman) has drafted a method for sampling light skins.

Subcommittee III on Physical Testing (G. A. Butz, chairman):

Section 2 on Physical Dimensions (E. Steiner, chairman), is developing a method of measuring thickness of soft leather and has presented an interim report of interlaboratory test data comparing thickness values of leathers of varied firmness measured by four methods, namely, Woburn gage, presser foot released gently; Woburn gage, presser foot snapped; Randall-Stickney gage, 200-g dead weight, and Randall-Stickney, 900-g dead weight.

Data analysis showed highly significant differences between values obtained on the Woburn when the gage foot is released gently and when it is snapped, on all but one very firm leather. The other method comparisons show differences too small to be of concern. The subcommittee will study thickness measurement comparisons on different leathers using the Woburn gage with snapping and the Randall-Stickney gage with various dead weight loadings.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] The new tentative appears in the 1961 Book of ASTM Standards, Part 6.

[2] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.

Section 3 on Tensile Properties (R. L. Young, chairman) has developed methods for double-hole slit tear resistance; Mullen test and extension; tensile strength; extension; and breaking strength and extension. Information on precision and accuracy is needed to complete the methods.

Data from tests using the single-hole stitch tear resistance method indicate the drilled hole is more precise than the punched hole. The results are as follows:

		Drilled	Punched
Standard Deviation	Pounds Tear...	16.8	22.0
	Pounds Tear per Inch.....	78.5	97.8
Coefficient of Variation	Pounds Tear...	13.5	18.0
	Pounds Tear per Inch.....	13.7	18.5

These results are based on tests made at one laboratory (the results of tests made by another laboratory are not complete). The method will contain a note referring to the punched hole.

Data from tests using the double-hole stitch tear resistance method which were made with three different diameter wires and two types of wire show very good correlation between three laboratories. The diameter and type wires tested were: 0.036 in. Type I, Size 1, Class A paper clips; 0.038 in. piano wire, and 0.041 in. Type I, Size 1, Class A paper clips. There was no significant difference in the results. On the data available, the diameter of the wire in the method will be 0.038 ± 0.003 in.

Data obtained on seven types of leather will be processed by a statistician at the National Bureau of Standards. Two other types of leather, welting and belting leather, will be tested and this will complete the testing to determine the reliability of the methods. After the data are evaluated, the precision-and-accuracy paragraph will be written and the methods submitted for approval by the joint committee.

Section 4 on Measurement of Water Resistance (R. G. Ashcraft, chairman) has completed drafts of the spray test for garment leather, the dynamic water-resistance test by the Sylflex Tester, and the Maeser method for water resistance.

Data were presented on static water absorption obtained in an inter-laboratory test on seven different types of leather by both the Kubelka method and method D 1815 – 60 T. The committee decided not to consider the Kubelka method further since no advantage in precision could be gained and the apparatus is fragile and expensive.

Apparatus developed at the National Bureau of Standards for measuring the dynamic water resistance of heavy leather has been modified and sent to a cooperating laboratory with a duplicate set of samples for obtaining data on interlaboratory reproducibility.

Consideration was given to the proposed method 8121 of Federal Specification KK-L-311a, which employs the Tap Tester for measuring resistance to water penetration. Interlaboratory tests are in prospect.

Comparison testing of water-repellent boots by the Dow Corning Shoe Tester and the wading test as recommended by the Quartermaster Laboratory at Natick, Mass. will be continued.

The section will develop a method for measuring water vapor permeability but will first attempt to correlate published methods.

Section 5 on Surface Characteristics

(Robert Stubbings, chairman) prepared the Method for Cold-Crack Resistance of Upholstery Leather which is being recommended for publication as tentative, as noted earlier in this report. In the light of negative ballots registered when the method was submitted to the Joint Committee in 1960, the method was modified to allow the sample to be taped together with pressure sensitive tape as well as with staples. The other change prescribes the use of fresh specimens for testing at another temperature.

A draft of a method for flex testing of finish of upholstery leather has been submitted to the Editorial Committee.

A major topic for discussion is the measurement of scuff resistance. In a recent preliminary test of seven leathers, many different types of devices to measure scuff were employed. These devices may be grouped in four general categories—pendulum devices, modified crockmeters, turntable and needle, and abrasion testers.

The section is also working on the three following problems:

1. Lightfastness—further tests to attempt to correlate South Florida exposures with fadeometers will be carried out.

2. Crock—a proposed method based on the crockmeter will be submitted for consideration of the Section at the next meeting.

3. Bleeding—a preliminary series of tests of a relatively simple test will be carried out.

Section 7 on Mechanical Leathers (A. Hirsch, chairman) continued studies on a proposed method to determine area stability of leather on immersion in water and oil.

Section 8 on Deterioration (C. F. Dudley, chairman) has current projects to evaluate deterioration from perspiration, from heat exposure, and in dry cleaning and laundering.

The effect of dry cleaning on water repellency was tested by three laboratories using a spray test. The results obtained by two laboratories agreed within the expected variation for the spray test. To clarify the discrepancy of results from the third laboratory, additional samples will be dry cleaned and spray rated.

A draft of a method of test for colorfastness and transfer of color in the washing of leather is under consideration. To meet suggestions made by Subcommittee I, the precision statement is being amended to indicate the degree of observer error to be anticipated in the gray rating scale.

A draft of a method for measuring resistance of upper leather to artificial perspiration was revised to meet comments in the section and from Subcommittee I.

Exposure of leathers in the Quartermaster climatic chambers continues. The laboratory evaluation will be accelerated.

Section 10 on Miscellaneous Physical Properties (Milton Bailey, chairman) has evaluated the Navy's Universal Leather Tester. Tests on matched, treated and untreated sole leather showed that butyl treatments substantially improved abrasive and water resistance of the leather. Also, leathers commercially treated by the hot-dip butyl process performed better than solvent-treated leathers.

Work is also progressing on an upper leather visual evaluation system.

Subcommittee IV on Research (M. M. Baldwin, chairman) is continuing its program of informal talks of interest to the entire committee. At the subcommittee meeting in March, interesting talks were presented by Dr. O'Flaherty of the University of Cincinnati on "The

Evaluation of Synthetics Used as Leather Substitutes," and by Mr. Bailey of the U. S. Department of the Navy on "Properties of Impregnated Versus Standard Sole Leathers."

The Council of the American Leather Chemists' Assn. has concurred in the recommendation of the committee that some attention be devoted to chemical methods of leather testing. The committee will accordingly organize subcommittees for this purpose.

Subcommittee V, Editorial (T. C. Thorstensen, chairman) has concerned itself principally with the form and organization of test methods.

This report has been submitted to letter ballot of the committee, which consists of 70 members; 36 members returned their ballots, of whom 29 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. NAGHSKI,
Chairman.

M. BAILEY,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, the Joint Committee on Leather presented to the Society through the Administrative Committee on Standards the recommendation for publication as tentative of a Method of Test for Resistance to Wetting (Spray Test) of Garment-Type Leather. This recommendation was accepted by the Standards Committee on September 12, 1961, and the new tentative method appears in the 1961 Book of ASTM Standards, Part 6, bearing the designation D 1913 – 61 T.

REPORT OF COMMITTEE E-1
ON
METHODS OF TESTING*

Committee E-1 on Methods of Testing held two meetings during the year: in Philadelphia, Pa., on Dec. 1, 1960, and April 25, 1961. Twenty-six subcommittees and task groups met during the 1960 Annual Meeting of the Society in Atlantic City. Subcommittee 17 on Thermometers and Hydrometers met in New York on Nov. 30, 1960, and in Washington, D. C., on May 19, 1961. Meetings were held of 18 subcommittees and task groups during ASTM Committee Week in Cincinnati, Ohio, from Jan. 30 to Feb. 3, 1961.

Mr. W. J. McCoy was elected vice-chairman of Committee E-1 for the ensuing term of two years.

The following seven members of Committee E-1 were appointed to the committee during the year by the Board of Directors: H. F. Hedderich, Pittsburgh Testing Laborabory, 2-yr term; E. W. Kuenzi, U. S. Forest Products Laboratory, 3-yr term; R. P. Lathrop, Wiedemann Machine Co., 1-yr term; K. N. Mathes, General Electric Co., 3-yr term; W. H. Mayo, U. S. Steel Corp., 3-yr term; W. T. Reid, Battelle Memorial Inst., 3-yr term; B. L. Wilson, National Bureau of Standards, 3-yr term.

Task Group A on Distillation Tests (J. S. Fawcett, chairman) completed its assignment and has accordingly been discharged.

Subcommittee 20 on Ceramic Laboratory Apparatus (H. W. Ryland, chairman) has been discontinued as there are no immediate problems requiring attention.

A Panel Discussion on Temperature Measurement in the Missile and Space Field is being sponsored by Section 6 on Application of Thermocouples for Airframe and Space Vehicles (R. B. Clapper, chairman) of Subcommittee 32 on Thermocouples for Temperature Measurement at the 1961 Annual Meeting.

A set of Recommendations on Form of ASTM Methods of Test has been completed by the committee. Copies in separate reprint form are being distributed to all technical committees of the Society.

Recommendations Accepted by the Administrative Committee on Standards

Subsequent to the 1960 Annual Meeting, Committee E-1 presented to the Society, through the Administrative Committee on Standards, the following recommendations:

New Tentative:

Specification for Standard Atmospheres for Conditioning and Testing Materials (E 171 – 60 T), to replace part of Tentative Definition with Procedures Relating to Conditioning and Weathering (E 41 – 57 T)

Revision of Tentative Methods of Test for:

Verification of Testing Machines (E 4 – 57 T),

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

Brinell Hardness of Metallic Materials (E 10 – 58 T),

Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials (E 18 – 59 T), and

Rapid Indentation Hardness Testing of Metallic Materials (E 103 – 54 T).

Revision of Tentative Definitions of Terms Relating to:

Methods of Mechanical Testing (E 6 – 58 T), and

Conditioning and Weathering (E 41 – 57 T).

Tentative Revision of Standard:

Hardness Conversion Tables for Metals (E 140 – 58).

The above recommendations were accepted by the Standards Committee on September 29 and December 19, 1960. These new and revised tentatives are published in the 1960 Supplement to Book of ASTM Standards, Parts 3, 5, 7, and 9.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated in the following tentatives and their continuation as tentative:

Methods of Verification of Testing Machines (E 4 – 60 T):[1]

Section 17.—In the first sentence change the expression "verified at intervals of at least 12 months" to read "verified at intervals of 12 months."

Specifications for ASTM Hydrometers (E 100 – 59 T):[2]

Table I.—For hydrometers 111H to 120H change the stem diameter from "5.0 mm, min" to "4.0 mm, min."

TENTATIVE REVISION OF STANDARD

The committee recommends that the two new Saybolt Furol Viscosity Thermometers shown in the accompanying Table I[3] be published as tentative as a tentative revision of the Standard Specifications for ASTM Thermometers (**E 1 – 60**).[4]

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revision as indicated below of the Standard Specifications for ASTM Thermometers (**E 1 – 60**)[4] and accordingly asks for the necessary nine-tenths affirmative vote in order that the revisions may be referred to letter ballot of the Society:

Table I, Thermometers 9 C and 10 C.—In Footnote *e* omit the second sentence which reads as follows: "The thermometer is mounted on a ferrule (see Appendix to Specifications for Pensky-Martens Closed Flash Tester (ASTM Designation: E 134)," as these thermometers are no longer furnished with ferrules.

ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard with revision as indicated:

Tentative Definitions of Terms Relating to Methods of Mechanical Testing (E 6 – 60 T):[5]

Section 10.—Add the following new

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[1] 1958 Book of ASTM Standards, Parts 3, 4, 5, 6, 9, 10.

[2] 1959 Supplement to Book of ASTM Standards, Parts, 7 and 8.

[3] Not included here; it appears in the revised specifications, 1961 Book of ASTM Standards, Parts 4, 7, and 8.

[4] 1960 Supplement to Book of ASTM Standards, Parts 4, 7, and 8.

[5] 1960 Supplement to Book of ASTM Standards, Parts 3, 4, 5, 9, 10.

definition of "Least Count" as Section 10, renumbering the remaining sections accordingly:

Least Count.—The smallest change in indication which can customarily be determined and reported.

NOTE.—In machines with close graduations it may be the value of a graduation interval; with open graduations, or with magnifiers, for reading, it may be an estimated fraction, rarely as fine as one-tenth, of a graduated interval; and with verniers it is customarily the difference between the scale and vernier graduation measured in terms of scale units. If the indicating mechanism includes a stepped detent, the detent action may determine the least count.

Section 15.—Revise the definition of strain and Note 1, retaining Note 2 without change, to read as follows:

Strain.—A measure of the change, due to force, in the size or shape of a body referred to its original size or shape. Strain is a nondimensional quantity, but it is frequently expressed in inches per inch, etc.

NOTE 1.—Strains may be either linear (tensile strain or compressive strain), or angular (shear strain). In the ordinary tension, compression, or torsion test, it is usual to measure only one component of strain and to refer to this as "the strain." Linear strain is defined as the change per unit of length in an original linear dimension. Shear strain is defined as the tangent of the angular change between two lines originally perpendicular to each other.

Section 21.—Revise the definition of fatigue and the Note to read as follows:

Fatigue.—The phenomenon of fracture under repeated or fluctuating (nonrepetitive) stress (Note) having a maximum value generally less than the tensile strength of the material.

NOTE.—Fatigue may also be expressed in terms of repeated or fluctuating (non-repetitive) loads of deformation. Evaluation in terms of deformation is especially useful in describing fatigue under conditions where the elastic limit of the material is exceeded. (Deformation is the sum of elastic and plastic strains.) In the definitions that follow, the terms "repeated stress," "cyclic stress," or "fluctuating (nonrepetitive) stress" are intended to cover any of the three conditions, that is, stress, load, or strain.

Section 61.—Revise the definition of creep and Notes 1 and 2 to read as follows:

Creep.—The time-dependent part of the strain resulting from stress.

NOTE 1.—Elastic and instantaneous strains, and thermal expansion are excluded.

NOTE 2.—While constant-stress creep tests are desirable, the usual one is a constant load test. Where the total strain is small, the difference is negligible, but for large strain corrections must be made in results of constant load tests, or the fact that the value is reported for constant load tests should be noted.

Section 62.—Revise the definition of creep recovery and the Note to read as follows:

Creep Recovery.—The time-dependent portion of the decrease in strain following unloading of a creep specimen.

NOTE.—Elastic strains and thermal expansion are excluded.

Appendix.—Add the abbreviation "psi" to represent "pounds per square inch," and the abbreviation "ksi" to represent "thousand pounds per square inch" or "kips per square inch."

Revise the symbol for Poisson's Ratio to read as follows:

μ (mu) (preferred), or ν (nu)—Poisson's ratio

Method for Determination of Young's Modulus at Room Temperature (E 111 - 59 T):[6]

Section 3(b).—Revise Note 3 to read: "NOTE 3.—The accuracy required in the determination of Young's modulus will vary from product to product and should be stated in the product specifications."

Footnote 7.—Delete this footnote which defines "least count" and renumber Footnote 8 as 7.

Method of Test for Indentation Hardness of Metallic Materials by Portable Hardness Testers (E 110 - 55 T):[7]

Section 4(a).—Omit the second sentence.

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[6] 1958 Book of ASTM Standards, Parts 3, 4, 5, 6, 9.

[7] 1958 Book of ASTM Standards, Part 3.

Method for Determination of Shear Modulus at Room Temperature (E 143 - 59 T):[8]

Section 3(b).—Revise Note 4 to read: "NOTE 4.—The accuracy required in the determination of shear modulus will vary from product to product and should be stated in the product specifications."

Footnote 11.—Delete this footnote which defines "least count" and renumber Footnote 12 as 11.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Methods of Compression Testing of Metallic Materials (E 9 - 59 T),[8]
Method of Test for Brinell Hardness of Metallic Materials (E 10 - 60 T),[7]
Specifications for Sieves for Testing Purposes (Wire Cloth Sieves, Round-Hole and Square-Hole Screens or Sieves) (E 11 - 60 T),[5]
Methods of Test for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials (E 18 - 60 T),[8]
Method of Rapid Indentation Hardness Testing of Metallic Materials (E 103 - 60 T),[7]
Specifications for Weighing and Drying Apparatus for Microchemical Analysis (E 124 - 57 T),[9]
Method of Test for Maximum Pore Diameter and Permeability of Rigid Porous Filters for Laboratory Use (E 128 - 57 T),[10]
Method for Determination of Poisson's Ratio at Room Temperature (E 132 - 58 T),[6]
Specifications for Apparatus for Microdetermination of Nitrogen by Kjeldahl Method (E 147 - 59 T),[11] and
Specifications for Apparatus for Microdetermination of Nitrogen by Dumas Method (E 148 - 59 T).[11]

ADOPTION OF TENTATIVE REVISION AS STANDARD

The committee recommends that the following twelve tentative Kinematic Viscosity Thermometer specifications in the Standard Specifications for ASTM Thermometers (**E 1 - 60**)[4] be adopted as standard: 28F, 29F, 30F, 43F, 44F, 45F, 46F, 47F, 48F, 72F, 73F, and 74F.

CONTINUATION OF TENTATIVE REVISION OF STANDARD

The committee recommends that the tentative revision of the Standard Hardness Conversion Tables for Metals (Relationship Between Brinell Hardness, Diamond Pyramid Hardness, Rockwell Hardness, and Rockwell Superficial Hardness (E 140 - 58)),[7] issued September 29, 1960,[12] be continued as tentative as further supplementary changes are in preparation.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the other tentatives under its jurisdiction be continued as tentative without revision as future contemplated changes are under study.

The recommendations in this report have been submitted to letter ballot of the Society, the results of which will be reported at the Annual Meeting.[13]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee 1 on Calibration of Mechanical Testing Machines and Apparatus (B. L. Wilson, chairman).—The need for additional methods for the calibration of testing machines under 2000-lb capacity is under study by a task group under the chairmanship of D. C. Scott, Jr.

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[8] 1959 Supplement to Book of ASTM Standards, Part 3.

[9] 1958 Book of ASTM Standards, Parts 4, 7, and 9.

[10] 1958 Book of ASTM Standards, Parts 5 and 7.

[11] 1959 Supplement to Book of ASTM Standards, Part 7.

[12] 1960 Supplement to Book of ASTM Standards, Parts 1, 2, and 3.

[13] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Methods of verification of testing machines at rapid rates of straining are under study by a task group under the chairmanship of A. A. Moore. No suitable methods for dynamic calibration have been found by the group. The task group is being enlarged and the work will be continued.

The Task Group on Calibration and Verification of Extensometers (R. H. Heyer, chairman) has in preparation changes in Method E 83.

Subcommittee 2 on Effect of Speed in Mechanical Testing (L. K. Irwin, chairman).—This subcommittee has reviewed the Tentative Recommended Practice for Short-Time Elevated Temperature Tension Tests of Material (E 21 - 58 T) prepared by the Joint ASME-ASTM Committee on Effect of Temperature on the Properties of Metals. This recommended practice specifies a constant normal strain rate for metals of either 0.005 or 0.05 in. per in. per min up to the yield point. Since it appears that most testing machines and extensometer-recorder equipment currently in use do not measure a recorded load and strain accurately at strain rates of 0.05 in. per in. per min and faster, it was agreed to recommend to the Joint Committee that consideration be given to requiring that a strain rate of 0.005 in. per in. per min shall be used unless it can be demonstrated that higher strain rates have no effect on the yield point.

A need has been indicated for standard methods of calibrating testing machines and accessories under dynamic conditions. The subcommittee accordingly plans to study this to determine whether it is feasible to develop such methods for testing machines and strain-recorder systems with first attention being given to the latter.

Subcommittee 3 on Elastic Properties and Definitions on Mechanical Testing (G. R. Gohn, chairman).—A task group has been appointed under the chairmanship of a representative of the Joint ASME-ASTM Committee on Effect of Temperature on the Properties of Metals, with other representatives from both that committee and Subcommittee 3, to draft a method for determining Young's modulus at elevated temperatures.

The Task Group on Elastic Constants (J. A. Miller, chairman) recommended the adoption as standard of the Tentative Methods E 111, E 132, and E 143 as covered earlier in this report.

The Task Group on Review of Definitions (J. Quigley, chairman) prepared the revisions in the general Definitions of Terms Relating to Methods of Mechanical Testing (E 6 - 60 T). The committee is reviewing a number of definitions prepared by other Society committees and also the extensive list of Definitions Relating to Metals and Metalworking in the ASM Metals Handbook. This will result in additional terms being included in Tentative E 6.

Subcommittee 4 on Tension Testing (F. M. Howell, chairman).—A complete study is being made of the Methods of Tension Testing of Metallic Materials (E 8 - 57 T) in an attempt to obtain better agreement with the Federal Test Method 211-1 and the ASTM Methods and Definitions for Mechanical Testing of Steel Products (A 370 - 60 T).

During the year the members of this subcommittee reviewed twelve methods of tension testing prepared by other Society committees. A number of comments and suggestions received were submitted to the committees responsible for the methods.

Subcommittee 5 on Compression Testing (J. A. Miller, chairman).—This subcommittee recommended the adoption as standard of the Tentative Methods of Compression Testing of Metallic Materials (E 9 - 59 T).

This subcommittee reviewed eleven

methods for compression testing prepared by other Society committees. A number of suggestions received were submitted to the originating committees.

Subcommittee 6 on Indentation Hardness (R. H. Heyer, chairman).—An extensive round-robin test program on the Rockwell C Hardness versus Tensile Strength Conversions for Steel has been completed. This resulted in a recommendation to Committee A-1 on Steel of a revised table for inclusion in the Tentative Methods and Definitions for Mechanical Testing of Steel Products (A 370 - 60 T). A report covering this test program appears in the May issue of *Materials Research & Standards*. The several methods for hardness testing, E 10, E 18, E 103, and E 110 are being recommended for adoption as standard, some with minor changes.

The proposed Knoop hardness conversion values for nickel and high-nickel alloys have been rechecked, and these data with accompanying explanatory notes are being prepared for inclusion in the Hardness Conversion Tables (E 140 - 58). The Method of Test for Hardness of Sintered Metal Friction Materials (B 347 - 59 T), developed by Committee B-9 on Metal Powders and Metal Powder Products, were reviewed and a number of comments submitted.

This subcommittee has been keeping in close touch with the work of ISO/TC 17 on the subject of Indentation Hardness Tests.

Subcommittee 10 on Sieves (L. V. Judson, chairman).—The chairman of this subcommittee and another member, A. E. Reed, as members of ASA Sectional Committee Z23 on Sieves were U. S. delegates to subcommittee meetings of ISO/TC 24 on Sieves in 1957 and 1959 and are now preparing for another ISO meeting late in 1961. By attendance at these meetings the merits of the United States viewpoint on sieve standards are receiving international recognition. The Tentative Specifications for Sieves for Testing Purposes (E 11 - 60 T) are being recommended for adoption as standard.

Subcommittee 12 on Methods for Density (C. T. Collett, chairman).—This subcommittee has prepared a revision of the Definition of Terms Relating to Specific Gravity (E 12 - 27). After approval by letter ballot these revised definitions will be submitted through Committee E-1 to Committee E-8 on Nomenclature and Definitions.

Subcommittee 14 on Conditioning and Weathering (F. M. Gavan, chairman).—Mr. J. E. Norton of the Atlas Electric Devices Co. was elected secretary of the subcommittee.

A revision of the Specifications for Enclosures and Servicing Units for Tests Above and Below Room Temperature (D 1197 - 56) has been completed and will be submitted for publication after approval by letter ballot. Also, new methods for the carbon-arc apparatus and the conduction of natural light exposures under glass have been completed and are now the subject of final letter ballot.

The measurement of water vapor transmission through thin materials is being studied at the request of Committees C-16 on Thermal Insulating Materials, D-20 on Plastics, and E-6 on Methods of Testing Building Constructions.

This will result in a revision of Methods E 96. This subcommittee is interested in the use of the plastic chip, developed by Committee D-20 on Plastics, as a measure of radiation intensity in artificial weathering machines.

Subcommittee 16 on Thickness Measurement (S. B. Newman, chairman).—An extensive revision of the Tentative Methods of Test for Thickness of Solid Electrical Insulation (D 374 - 57 T) is being voted upon. The scope of these methods has been enlarged so that they can be used for measuring the thickness of solid pressure-sensitive materials. This includes not only electrical insulation, but also such materials as woven glass fabrics, rigid rods, vulcanized fiber, mica, paper, phenolic and other plastic sheets and laminates.

Subcommittee 17 on Thermometers and Hydrometers (J. F. Swindells, chairman).—Active cooperation with ISO/TC 48 is continuing. The subcommittee approved changes in certain dimensions for the hydrometer series 111H to 120H in Specifications E 100 which bring these dimensions in conformity with ISO proposals.

Specifications for two new Saybolt Furol viscosity thermometers for tests at 275 and 325 F requested by Committees D-4 and D-8 have been completed.

Tentative results from the first tests involving a comparison of the ASTM method for bulb stability with a proposed ISO test were reported by the National Bureau of Standards. No conclusions with respect to the relative merits of the two methods have been reached, and the tests are continuing.

A task group is making a study of the standardization temperatures specified for all ASTM thermometers. The results of this work will be important to both manufacturers and users alike, since adequate standardization and calibration costs are both involved.

The subcommittee sincerely regrets the withdrawal of Robert D. Thompson from its activities. Mr. Thompson has made many important contributions to the work of the subcommittee as a participating member, secretary, and chairman. His sound council will be greatly missed.

Subcommittee 19 on Glassware Laboratory Apparatus (J. J. Moran, chairman).—Working groups have been requested to prepare standard recommendations for drawings of glassware and to canvass the possibilities of standard specifications for stirrers for laboratory vessels.

Subcommittee 25 on Shear and Torsion Tests (R. W. Fenn, Jr., chairman).—A task group under the chairmanship of R. B. Clapper has been appointed to study various shear testing techniques and to write recommended shear test procedures for consideration by the subcommittee. Task group members are actively engaged in research on various types of shear tests aimed at defining the advantages, disadvantages, and limitations of each. Consideration is being given to standardization of: (1) a new double-shear test for determining longitudinal and transverse properties of sheet materials, (2) a punch shear test for determining short transverse properties of sheet materials, (3) a pin double-shear test for use on bar, rod, and plate materials, (4) a new method of determining shear strength of adhesive bonded materials, and (5) a shear test for sandwich constructions. Photoelastic studies of certain test specimen configurations are being made in an effort to determine the degree of pure shear in the shear path. Although the task group is working diligently on the above tests, there are several problems to be solved before recommended procedures can be submitted for committee approval.

Subcommittee 26 on Filtration Materials (T. L. King, chairman).—This subcommittee recommended the adoption as standard of the Tentative Method of Test for Maximum Pore Diameter and Permeability of Rigid Porous Filters (E 128 – 57 T). Plans will be initiated at the June meeting on an inter-laboratory test program to study the reproducibility of this method.

Subcommittee 27 on Low-Temperature Testing of Elastomers and Plastics (F. M. Gavan, chairman).—A summary of the low-temperature studies by this subcommittee was published in a report on "Rheological Testing of Elastomers at Low Temperatures, Part II," by Boor, Hanok, Conant, and Scoville which appeared in the ASTM BULLETIN No. 246, May, 1960. A complete summary of the extensive cooperative studies made by this subcommittee is in preparation.

Subcommittee 28 on Microscopy (T. G. Rochow, chairman).—A list of definitions of terms relating to microscopy prepared by the Task Group on Nomenclature (H. F. McMurdie, chairman) will be issued as tentative after the Annual Meeting.

The subcommittee is planning to endorse the American Standard Microscope Objective Thread (ASA B-1.11-1958) and wishes to arrange for the publication of this by ASTM. Specifications and Methods of Test for Glass Covers and Slides Used in Microscopy are being completed by the Task Group on Microscopes and Their Elements (O. W. Richards, chairman). A Bibliography on Microscopy, prepared by the Task Group on ASTM Bibliography (R. P. Loveland, chairman) will be ready for publication this summer. A Symposium on the Preparation of Materials for Microscopic Examination is being planned by the Task Group on Preparation of Materials (Katharine Mather, chairman) for the 1962 Annual Meeting.

A session on non-commercial microscopic accessories will be held during the Annual Meeting.

Subcommittee 29 on Microchemical Apparatus (A. Steyermark, chairman).—Specifications are being prepared for apparatus used in connection with micro-analytical determination of carbon and hydrogen by the conventional method of Pregal. The three existing tentative specifications for microchemical apparatus are being recommended for adoption as standard.

Subcommittee 30 on Bend Test for Ductility (W. H. Mayo, chairman).—A new method for guided bend test for metallic materials has been completed and will be submitted for publication as tentative later in the year. Work is being undertaken on a general bend test specification for steel products to contain semi-guided and wrap-around procedures.

Subcommittee 31 on Laboratory Balances (L. B. Macurdy, chairman).—This subcommittee has in preparation definitions of a number of terms and is also developing a classification of balances and scales with information on typical applications. Performance specifications for analytical balances are also under study.

Subcommittee 32 on Thermocouples for Temperature Measurement (J. W. Freeman, chairman).—This subcommittee has several active sections on the following subjects:

1. Standardization of Standard Thermocouple Materials (D. I. Finch, chairman),
2. Newer Thermocouple Materials (A. I. Dahl, chairman),
3. Review of ASTM Standards and Thermocouples in Nuclear Environment (J. G. Lachman, chairman),
4. Monograph on Use of Thermocouples (J. W. Murdock, chairman),
5. Evaluation of Thermocouple Materials (C. D. Starr, chairman),
6. Application of Thermocouples for Airframe and Space Vehicles (R. B. Clapper, chairman), and
7. Insulating Materials for Thermocouples.

A number of tentative methods and recommended practices are being prepared by these sections. It is expected that several will be completed for publication in 1962.

A review of the application of thermocouples in ASTM Standards is nearing completion.

Task Group B on Linear Thermal Expansion Test (H. E. Hagy, chairman).—This newly organized task group has undertaken the development of an all-inclusive method for rigid solids based on the use of the fused quartz dilatometer because of its popularity and reliability. This procedure will cover a temperature range from 0 to 800 C or possibly to 1000 C. A literature survey is being made on high-temperature methods (1500 C and over).

This report has been submitted to letter ballot of the committee and its subcommittee chairmen, a total of 40 voting members; 32 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

A. C. WEBBER,
Chairman.

P. J. SMITH,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specifications for:

Apparatus for Microdetermination of Carbon and Hydrogen (E 191 – 62 T).

New Tentative Method for:

Guided Bend Test for Ductility of Welds (E 190 – 61 T).

New Tentative Definitions of Terms Relating to:

Microscopy (E 175 – 61 T).

New Tentative Recommended Practice for:

Conducting Natural Light (Sunlight and Daylight) Exposures Under Glass (E 187 – 61 T), and

Operation of Enclosed Carbon Arc Type Apparatus for Artificial Light Exposure Tests (E 188 – 61 T).

Revision of Tentative Methods of:

Tension Testing of Metallic Materials (E 8 – 57 T).

Revision and Reversion to Tentative of:

Standard Specifications for Enclosures and Servicing Units for Tests Above and Below Room Temperature (D 1197 – 56), redesignated E 197 – 61 T, and

Standard Definitions of Terms Relating to Specific Gravity (E 12 – 57).

Tentative Revision of Standard Definitions of Terms Relating to:

Methods of Mechanical Testing (E 6 – 61).

The new Tentative Specifications E 191 – 62 T were accepted by the Standards Committee on March 8, 1962 and are available as a separate reprint. The other recommendations were accepted on September 12, 1961, and appear in the 1961 Book of ASTM Standards.

REPORT OF COMMITTEE E-2

ON

EMISSION SPECTROSCOPY*

Committee E-2 on Emission Spectroscopy held two meetings during the year: in Atlantic City, N. J. on June 29, 1960, and in Pittsburgh, Pa., on March 2, 1961. The Executive Subcommittee also met at these times as well as in New York, N. Y., on Nov. 2, 1960.

A main activity of the committee was the preparation of the third revised edition of the book entitled "Methods for Emission Spectrochemical Analysis" which was published in March, 1961. Also published in March, 1961, was *ASTM STP No. 58-D* entitled "Report on Available Standard Samples and Related Materials for Spectrochemical Analysis." This is the fourth revision of the original publication and replaces the 1955 edition.

Published in June, 1960, was Part IV of the "Index to the Literature on Spectrochemical Analysis." This covers the years 1951 to 1955 and was issued as *ASTM STP No. 41-D.*

A new publication, "Index to the Literature on X-Ray Spectrographic Analysis" was prepared and published in January, 1961. This is Part I covering the years 1913 to 1957, and was issued as *ASTM STP No. 292.*

A Symposium on Extension of Sensitivity has been planned jointly with Committee E-3 on Chemical Analysis to be held June 28, 1961, at the Annual Meeting in Atlantic City, N. J.[1]

The committee has 210 members, which is an increase of six members since the last Annual Meeting. The official voting membership is 160. Efforts continuously are being made to obtain active members, particularly from the western part of the United States and from Canada.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1960 Annual Meeting, Committee E-2 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Recommended Practice for:

Describing and Specifying the Excitation Source in Emission Spectrochemical Analysis (**E 172 – 60 T**).

Revisions of Tentative Definitions of:

Terms and Symbols Relating to Emission Spectroscopy (**E 135 – 60 T**).

The new Recommended Practice E 172 was accepted by the Standards Committee on September 29, 1960, and the revisions of Definitions E 135 were accepted on September 16 and September 29, 1960. Both are available as separate reprints and appear in the 1960 Book of Methods for Emission Spectrochemical Analysis and the 1960 Book of ASTM Methods for Chemical Analysis of Metals.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] To be published as *ASTM STP No. 308.*

SUGGESTED METHODS TO BE PUBLISHED AS INFORMATION

The committee recommends for publication as information only the following Suggested Methods:[2]

Suggested Methods for Spectrochemical Analysis of:

Indium-Gallium Alloys by the Solution-Graphite Spark Technique (SM 6-21), and

Blast Furnace and Steelmaking Slags by the Fusion Spark Technique (SM 11-12)

ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that the Tentative Method for Spectrochemical Analysis of Nickel Alloys by the Powder—D-C Arc Technique (**E 129 - 57 T**)[3] be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

Title.—Change to read "Method for Spectrochemical Analyses of Thermionic Nickel Alloys by the Powder - D-C Arc Technique."

Footnote 4.—Delete the last phrase "and is marketed by Spex Industries, Inc., Queens Village, N.Y."

Section 1.—Change Paragraph (*a*) to read "This method provides for the determination of the following elements in thermionic nickel alloys in the concentration ranges shown." In the table, delete lead, zinc, and boron.

Add a new Paragraph (*c*) to read

> (*c*) The concentration ranges of the elements listed in Paragraph (*a*) have been established through cooperative testing of secondary standards and by preparation of synthetic standards, provided in the method. The scope is underwritten by three primary spectrochemical standards.

Section 5.—Delete Paragraph (*a*) and reletter subsequent paragraphs accordingly.

Section 6.—Change to read: "Three primary standards are available for use with this procedure. Additional standards can be prepared in the laboratory to supplement the scope, if desired."

Section 7.—Reletter present Paragraph (*a*) as (*b*), and delete references to boron.

Delete present Paragraph (*b*).

Add a new Paragraph (*a*) to read as follows:

> 7. (*a*) *Primary Standards.*—Disolve 0.3 g or more of the primary standards in HNO_3 (1:1). Evaporate the solution to dryness in a platinum evaporating dish, bake at 400 C for 20 min, grind, and mix thoroughly with a suitable mortar and pestle. Mix the dry powder with three parts by weight of graphite powder.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Recommended Practices for:

Photographic Processing in Spectrochemical Analysis (**E 115 - 59 T**),

Photographic Photometry in Spectrochemical Analysis (**E 116 - 59 T**), and

Designation of Shapes and Sizes of Preformed Graphite Electrodes (**E 130 - 57 T**).

Tentative Methods for Spectrochemical Analysis of:

Zinc-Base Alloys and High-Grade Zinc by the Solution Residue—D-C Arc Technique (**E 27 - 59 T**),

Tin Alloys for Minor Constituents and Impurities (**E 51 - 59 T**),

Aluminum and Aluminum-Base Alloys by the Point - to - Plane Spark Technique (**E 101 - 53 T**), and

Pig Lead by the Point-to-Plane Spark Technique (**E 117 - 59 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

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[2] See pp. 620 and 624.

[3] 1960 Book of ASTM Methods for Chemical Analysis of Metals.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Apparatus and Equipment (A. W. Helz, chairman) has prepared the Tentative Recommended Practice for Describing and Specifying the Excitation Source in Emission Spectrochemical Analysis as referred to earlier in this report. The subcommittee is working on (1) photographic emulsions and has planned publications for the suggested practice for the description and specification of photographic emulsions, report on available photographic emulsions, and report on the test program of SA No. 1 cut film; (2) description and specification of the emission spectrograph; (3) description of the X-ray spectrograph; and (4) description and specifications of the microphotometer.

Subcommittee II on Fundamental Practices (J. K. Hurwitz, chairman) has prepared a format, in cooperation with Subcommittee III and, after revision, adopted a model method for writing methods on X-ray spectrochemical analysis. The subcommittee has prepared and published a bibliography on X-ray spectroscopy covering the years 1913 to 1957 as described earlier in this report. The preparation and publication of Part II of the bibliography has been planned. The subcommittee also has published a list of unsolved problems in spectrochemical analysis, and is planning the publication of reports on the status of the unsolved problems. Study and evaluation is being made of the suggested practice for use of statistical methods in spectrochemical analysis for elevation to tentative status.

Subcommittee III, Editorial (D. C. Spindler, chairman) has (1) proposed several changes in the Regulations for E-2 and Recommendations for Preparation of E-2 Practices and Methods; (2) reviewed proof for the new edition of "Methods for Emission Spectrochemical Analysis"; (3) made editorial review of 24 practices and methods; (4) cooperated with Subcommittee II in the preparation of a format and model method for X-ray spectrochemical analysis; and (5) revised Tentative Definitions E 135 referred to earlier in this report.

Subcommittee IV on Electrodes, Pure Materials, Reagents, and Standards (W. R. Kennedy, chairman) has prepared the 1960 "Report on Available Standard Samples and Related Materials for Spectrochemical Analysis" referred to earlier. The subcommittee is (1) revising the tentative designation of recommended shapes and sizes of preformed graphite electrodes; (2) testing the physical and chemical properties of graphite and carbon electrodes; (3) surveying methods for the preparation of homogeneous metals, alloys, and powders; and (4) obtaining a listing of suitable line pairs for a variety of matrices.

Subcommittee V on Copper, Nickel, Rhenium and Their Alloys (R. S. Kelly, chairman) is (1) cooperating with the National Bureau of Standards in the homogeneity testing of proposed copper-base and high-temperature alloy spectroscopic standards; (2) planning a symposium on the spectroscopic analysis of high-temperature alloys; (3) performing cooperative test programs with several suggested methods for tentative status; and (4) writing new X-ray spectrochemical methods. By mutual agreement with Subcommittee IX, Subcommittee V will be responsible for iron, as well as nickel- and cobalt-base high-temperature alloys, except the general classification of stainless and super stainless steels. The subcommittee recommended that Method E 129 be adopted as standard with revision as noted earlier in this report.

Subcommittee VI on Lead, Tin, Zinc, Indium, and Related Metals (J. F.

Murphy, chairman) (1) prepared for publication two suggested methods on indium alloys (SM 6-20 and SM 6-21), and is processing four additional methods on indium; (2) is working on cooperative test programs to bring four suggested methods to tentative status: SM 6-9 on tin and SM 6-12, SM 6-15, and SM 6-18 on zinc; (3) is surveying the membership and elsewhere for X-ray spectrochemical methods; and (4) is investigating the possibility of a symposium with other subcommittees and groups on various analytical techniques related to the semiconductor industry, such as for gallium, germanium, indium, and gold.

Subcommittee VII on Aluminum, Magnesium, Gallium and Their Alloys (F. R. Potter, chairman) has prepared for publication two suggested methods on aluminum (SM 7-14 and SM 7-15). The subcommittee is (1) processing data from a cooperative test program on a spectrometric method for aluminum and its alloys (SM 7-10) for advancement to tentative; (2) processing three suggested methods on magnesium for publication; and (3) establishing task groups for work on gallium and gallium alloys, analyses of trace constituents in aluminum, and microdrill sampling techniques as applied to aluminum.

Subcommittee VIII on Titanium, Zirconium, Molybdenum, Tungsten, Niobium, Tantalum, Beryllium, and Related Alloys (N. E. Gordon, chairman) has prepared for publication one suggested method for titanium (SM 8-11) and two for zirconium (SM 8-12 and SM 8-13). The subcommittee is (1) assisting the National Bureau of Standards in the homogeneity testing and analysis of six proposed zirconium-base standards; (2) engaged in cooperative test programs for zirconium methods; and (3) actively soliciting emission spectrochemical methods of analysis for the refractory elements and their alloys.

Subcommittee IX on Ferrous Alloys (J. F. Woodruff, chairman) has published four suggested methods (SM 9-19, SM 9-20, SM 9-21, and SM 9-22). This subcommittee has 20 active task groups, seven of which are performing cooperative test programs to support the acceptance as tentative of the following suggested methods: SM 9-11, SM 9-12, SM 9-13, SM 9-14, SM 9-16, SM 9-18, and SM 9-21. In addition, several new suggested methods are being processed for publication, and X-ray methods are being solicited.

Subcommittee X on Glass, Ceramics, Alkalies, and Cementitious Materials (W. M. Hazel, chairman) has prepared for publication two suggested methods: one for alumina (SM 10-18) and one for alkalies in titanium, aluminum, and zirconium oxides (SM 10-19). It is (1) processing three suggested X-ray spectrochemical methods for publication, with two additional X-ray methods to be submitted; and (2) maintaining close liaison with the interested technical committees to supply their analytical needs in spectrochemical analysis.

Subcommittee XI on Slags, Ores, and Other Miscellaneous Nonmetallic Materials (R. G. Russell, chairman) has published three suggested methods: one on blast-furnace and open-hearth sinter (SM 11-10), one on rare earths and thorium in cerium minerals (SM 11-11), and one for blast-furnace and steelmaking slags (SM 11-12). The subcommittee is (1) planning a cooperative test program for the suggested method on blast-furnace and steelmaking slags; (2) soliciting X-ray spectrochemical methods of analysis; and (3) establishing close liaison with the C and D Committees to ensure adequate coverage by spectrochemical analysis to meet the analytical needs for the materials under their jurisdiction.

This report has been submitted to letter ballot of the committee, which consists of 210 members; 141 members returned their ballots of whom 135 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

ROBERT E. MICHAELIS,
Chairman.

DAVID W. HENTHORN,
Secretary.

APPENDIX I

SUGGESTED METHOD FOR SPECTROCHEMICAL ANALYSIS OF INDIUM-GALLIUM ALLOYS BY THE SOLUTION-GRAPHITE SPARK TECHNIQUE[1]

SUBMITTED BY E. M. MURT[2] AND I. CORSON[2]

E-2 SM 6-21

Scope

1. (*a*) This method covers the determination of gallium in the range from 0.1 to 1.2 per cent in indium-gallium alloys.

(*b*) The method is designed for the routine testing of individual indium-gallium pellets or spheres used in transistor production and development. The method is limited by the inconvenience of handling samples weighing less than 0.040 mg and the requirement of making three determinations on each sample. Each determination requires 0.010 mg of alloy.

Summary of Method

2. This method is based on the method described by E. M. Murt and J. C. Bready[3] for the determination of gallium in micro-samples of indium-gallium alloys, but has been revised to use indium as the internal standard. A controlled spark discharge supplies the excitation between two flat-surface electrodes[4] on which the solution has been dried. The spectra are recorded photographically. The intensity ratio of the gallium analytical line and the indium internal standard line is determined photometrically, and the concentration of the gallium is read from an analytical curve relating log intensity ratio to log concentration.

Apparatus

3. (*a*) *Excitation Source.*—Excitation is obtained by a spark controlled by an auxiliary air gap.

(*b*) *Spectrograph.*—A large Littrow-type quartz prism spectrograph, having a reciprocal linear dispersion of 7 A per mm at 3400 A and 2 A per mm at 2200 A, is employed. The distance from the spectrograph slit to the center of the spark gap is 61 cm. A quartz lens is employed to form an enlarged image of the light source on the slit. A two-step filter with transmittances of 100 and 10 per cent is used in the light path to reduce the spectral intensity of the indium internal standard line.

(*c*) *Photographic Processing Equipment*, providing developing, fixing, washing, and drying operations and conforming to the requirements of the Recommended Practices for Photographic Processing in Spectrochemical Analysis (ASTM Designation: E 115).[5]

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[1] **This suggested method has no official status in the Society but is published as information only. The method is based on the experience of the submitters. Comments are solicited.**

[2] Philco Corp., Lansdale Division, Lansdale, Pa.

[3] E. M. Murt and J. C. Bready, "The Quantitative Determination of Gallium in Micro Samples by the Graphite Spark," *Applied Spectroscopy*, Vol. 15, No. 1, p. 1 (1961).

[4] **J. M. Morris and F. X. Pink, "Trace Analysis by Means of the Graphite Spark,"** Symposium on Spectrochemical Analysis for Trace Elements, *ASTM STP No. 221*, Am. Soc. Testing Mats. (1958).

(*d*) *Microphotometer.*—The spectral lines are measured with a nonrecording projection densitometer incorporating a barrier-layer type photocell and a galvanometer. It shall be suitable for the measurement of transmittances of spectral lines with a standard deviation of approximately 0.2 per cent transmittance.

(*e*) *Calculating Equipment.*—A calculating board with emulsion calibration scales or curve to convert microphotometer readings to intensity ratios.

(*f*) *Precision Torsion Balance,*[6] with a sensitivity of 0.002 mg.

Reagents and Materials

4. (*a*) *Purity and Concentration of Reagents.*—The purity and concentrations of the chemical reagents used in preparing the standards and samples shall conform to the requirements prescribed in the Recommended Practices for Apparatus and Reagents for Chemical Analysis of Metals (ASTM Designation: E 50).[7]

(*b*) *Apiezon N Solution.*[8]—Dissolve 5.0 g of Apiezon N in 1 liter of petroleum ether (certified, ACS, bp 30 to 60 C, sp gr 0.625 to 0.650/25 C).

(*c*) *Electrodes.*—High-purity graphite rods as described in Section 7.

(*d*) *Gallium Stock Solution* (*0.1 mg Ga per ml*).—Dissolve 10 mg of high-purity gallium in 10 ml of HCl and make up to 100 ml with water in a volumetric flask.

(*e*) *Indium Stock Solution* (*1.0 mg In per ml*).—Dissolve 100 mg of high-purity indium in 10 ml of HCl and make up to 100 ml with water in a volumetric flask.

(*f*) *Photographic Emulsion.*—Eastman No. 103-0.

(*g*) *Photographic Processing Solutions.*—The formulas for photographic Processing solutions are given in Recommended Practices E 115.

Preparation of Standards

5. Use the stock solutions (Section 4 (*d*) and (*e*)) to prepare a series of standard solutions to cover the gallium range from 0.1 per cent to 1.2 per cent. Prepare the standards on the basis of 0.10 mg of total metal per milliliter of solution. Adjust the acid concentration of the standards to approximately 2 *N*.

Preparation of Samples

6. Weigh samples of the indium-gallium pellets or spheres and transfer each individual alloy to a volumetric flask. Transfer samples weighing between 0.04 and 0.10 mg to a 1-ml volumetric flask and add 0.1 ml to 0.2 ml of 8 *N* HCl. Heat the sample slowly if it does not readily dissolve. After the sample has dissolved, dilute to give a final alloy concentration of 0.10 mg In-Ga per ml of approximately 2 *N* HCl. For samples weighing more than 0.10 mg, it is necessary to use larger volumetric equipment and a proportionately larger amount of acid. Individual sample spheres do not normally weigh more than 50 mg. It is recommended that a final concentration of 0.10 mg of sample per ml be obtained if possible.

Electrode System

7. The upper and lower electrodes shall be high-purity graphite rods, $\frac{3}{16}$ in. in diameter, and 1.5 in. in length. The flat surface tips of the upper and lower

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[5] "Methods for Emission Spectrochemical Analysis," Am. Soc. Testing Mats., Third Edition (1960).

[6] The balance manufactured by the Bethlehem Instrument Co., Inc., Bethlehem, Pa., has been found satisfactory for this purpose.

[7] 1960 Book of ASTM Methods for Chemical Analysis of Metals.

[8] Available from James G. Biddle Co., 1316 Arch St., Philadelphia 7, Pa.

electrodes, to which the sample is to be added, should be coated with two drops of Apiezon N solution. Allow the first drop of Apiezon N to dry before adding the second drop. Pipet 0.05 ml of the sample solution onto each of the electrodes. Allow the solutions to dry on the electrode surface under an infrared lamp. Store the electrodes with the dried samples under the infrared lamp until ready to be sparked. Adjust the analytical gap to 2.0 mm.

TABLE I.—ANALYTICAL LINE PAIRS.

Element	Analytical Line, A	Excitation Potential, ev	Concentration Range, per cent	Concentration Index, per cent
Gallium	Ga I 2874.24	4.29	0.1 to 1.2	0.3
Indium	In I 2932.62[a]	4.47	internal standard	...

[a] The intensity of this line is reduced by a 10 per cent transmittance filter.

Excitation and Exposure

8. Produce and record the spectra according to the following conditions:

(*a*) *Electrical Parameters.*—The constants read from the meters and dials of a commercially available source unit[9] are as follows:

Discharge voltage, v	8000
Capacitance, μf	0.005
Inductance, μh	600
Secondary resistance, ohm	3
Radio frequency current, amp	2.0
Number of discharges per cycle	14

(*b*) *Exposure Conditions:*

Spectral region, A	2490 to 3470
Slit width, mm	0.030
Slit height, mm	4
Spark preburn period, sec	0
Spark exposure period, sec	20
Filters	as noted in Table I and Section 3(*b*)

(*c*) *Exposure Index.*—Maintain the transmittance of the In I 2932.62 A line at approximately 60 per cent.

(*d*) *Replicate Exposures.*—Make triplicate exposures for each sample along with two or more control standards in duplicate.

Photographic Processing

9. Process the emulsion in accordance with the Recommended Practices E 115.

Photometry

10. Measure the percentage of transmittance of the analytical line pair with a projection microphotometer. The analytical line pair is shown in Table I.

Calibration

11. (*a*) *Emulsion Calibration.*—Calibrate the emulsion in accordance with the Recommended Practices for Photographic Photometry in Spectrochemical Analysis (ASTM Designation: E 116).[5]

(*b*) *Preparation of Analytical Curve.*—Read and record transmittance measurements of two spectra for each of five standard samples that cover the test range, on at least three different plates, to obtain the original analytical data. Convert the transmittance measurements of the analytical line and the internal standard line to log-intensity ratios, using the emulsion calibration curve. Prepare an analytical curve by plotting the log intensity ratio versus the log concentration using the average of six determinations for each point on the curve.

(*c*) *Analytical Curve Control.*—Make duplicate exposures of three control duplicate exposures of two or more control standards on each plate to check the original analytical curve and to determine corrections when necessary.

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[9] The specific settings given here are for the Spec-Power Unit Model J-12-22, manufactured by the National Spectrographic Laboratories, Inc., Cleveland, Ohio.

TABLE II.—DATA ON PRECISION OF METHOD.

Element	Average Concentration, per cent	Coefficient of Variation[a]	Number of Determinations
Gallium	0.19	16.3	10
	0.61	12.6	10
	0.95	9.9	10

[a] Coefficient of variation, ν, in this method, is calculated as follows:

$$\nu = \frac{100}{\bar{X}} \sqrt{\frac{\Sigma d^2}{n - 1}}$$

where:

$\bar{X}$ = average concentration, in per cent,

d = deviation of the determination from the mean, and

n = number of determinations.

Calculations

12. Convert the transmittances of the analytical and the internal standard lines of the sample into log intensity ratios. Determine the percentage concentration from the previously prepared analytical curve. Report the average of the triplicate determinations for each sample.

Precision and Accuracy

13. (*a*) *Precision.*—The data on precision of this method are shown in Table II.

(*b*) *Accuracy.*—The accuracy of the method is dependent on the reliability of the solution standards. It is estimated that the accuracy of the method is comparable to its precision.

DISCUSSION

The method should be applicable to other elements in indium alloys besides gallium when the proper solution standards have been prepared. This method offers a means of checking individual pellets or spheres even though they weigh considerably less than 0.1 mg. It can aid in the study of segregation effects, processing variables, and can be applied where the sample size is limited for indium-gallium alloys.

APPENDIX II

SUGGESTED METHOD FOR SPECTROCHEMICAL ANALYSIS OF BLAST FURNACE AND STEELMAKING SLAGS BY THE FUSION SPARK TECHNIQUE[1]

SUBMITTED BY P. R. IRISH,[2] H. VELDHUIS,[3] AND E. H. LUCAS[4]

E-2 SM 11-12

Scope

1. (*a*) This method covers the spectrochemical analysis of blast furnace and steelmaking slags for the following constituents in the concentration ranges shown:

Constituent	Concentration Range, per cent
Calcium oxide (CaO)	15.0 to 60.0
Silicon dioxide (SiO_2)	7.0 to 50.0
Ferrous oxide (FeO)	15.0 to 35.0
Magnesium oxide (MgO)	4.0 to 25.0
Aluminum oxide (Al_2O_3)	1.00 to 20.0
Titanium dioxide (TiO_2)	0.5 to 10.0
Manganous oxide (MnO)	0.3 to 15.0

(*b*) This method can be extended in scope with slight modification to cover ores, refractories, and other inorganic substances. The method is similar in scope to the Suggested Method for Spectrochemical Analysis of Slags by the Powder - D-C Arc Technique (E-2 SM 11-6),[5] but provides for determinations at higher concentrations of titanium dioxide and magnesium oxide and at lower concentrations of manganous oxide. The method is broader in scope than the Suggested Method for Spectrochemical Analysis of Open-Hearth Slags for Lime-Silica Ratios by the Powder - Intermittent Arc Technique (E-2 SM 11-7),[5] and the Suggested Method for Spectrochemical Analysis of Basic Open-Hearth Slags for Lime-Silica Ratios by the Powder - Spark Technique (E-2 SM 11-8).[5]

Summary of Method

2. A weighed sample of powdered slag is fused with a flux that contains an internal standard and a buffer material. One end of a short graphite rod is dipped into the molten material. When the rod is removed, a cap of the melt adheres (Note 1). This rod and one uncoated electrode constitute an electrode pair which is subjected to spark excitation. The spectra are photographed and the intensity ratios of selected pairs of analytical and internal standard lines are determined photometrically. The concentrations of the constituents sought are obtained from analytical calibrations which relate log intensity ratio to per cent concentration.

NOTE 1.—The technique of dipping the graphite rod into the molten material is a critical operation affecting the accuracy of the method.

Apparatus

3. (*a*) *Sample Preparation Equipment:*

(*1*) *Blender.*—Any suitable apparatus, such as a sealed jar mounted on a lathe and rotated at a moderate speed (Note 2).

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[1] This suggested method has no official status in the Society but is published as information only. The method is based on the experience of the submitters. Comments are solicited.

[2] Bethlehem Steel Co., Bethlehem, Pa.

[3] Ford Motor Co., Dearborn, Mich.

[4] American Steel and Wire Div., United States Steel Corp., Worcester, Mass.

[5] "Methods for Emission Spectrochemical Analysis," Am. Soc. Testing Mats., 3rd Ed. (1960).

NOTE 2.—Large quantities (2000 g) of flux may be mixed in a commercial apparatus.[6]

(*2*) *Fusion Furnace.*—A 2-kva induction furnace adjusted so that within 3 min the temperature of the melt will be 1150 C.[7]

(*3*) *Dipping Clamp.*—A rod fitted with a tapered adapter to hold the electrodes. This rod shall be suspended vertically and directly above the furnace opening by means of a cross arm and friction clamps on a support. This apparatus shall be used to lower and raise the electrodes in the dipping process.

(*4*) *Sample Storage Blocks.*—Carbon or ceramic blocks approximately 2 by 6 by 0.5 in. shall be drilled with rows of holes $\frac{9}{32}$ in. in diameter to a depth of $\frac{1}{4}$ in. Other dimensions may be found satisfactory.

(*5*) *Drying Oven or Infrared Lamps,* for keeping the dipped electrodes dry during storage.

(*b*) *Excitation Source.*—Excitation is obtained by a high-voltage controlled spark discharge. Either the air-interrupted spark source[8] having the parameters specified in Section 8 (*a*) or the rectified spark with rotary interrupter[9] may be used. Proper parameters shall be employed with either type source to produce desired precision and accuracy.

(*c*) *Spectrograph,* capable of covering the wavelength region 2500 to 3600 A. The linear dispersion and resolving power shall be great enough to separate Mn 2933.06 A and Cr 2932.70 A in the spectrum of a slag sample containing a minimum of 2.0 per cent manganese and 1.0 per cent chromium. These conditions are satisfied by grating and quartz prism spectrographs having a reciprocal linear dispersion of 6 A per mm at 3200 A. The spectrograph, with external optics, shall produce sharp lines of uniform density (Note 3). The illumination of the spectrograph shall represent radiation from all parts of the spark discharge (Note 4).

NOTE 3.—In the case of a quartz Littrow instrument, better uniformity of the spectral lines can be obtained by placing a lens directly before the slit to image the source upon the collimating lens.

NOTE 4.—Direct - reading spectrometers having the required resolving power are adaptable to this procedure.

(*d*) *Pin Specimen Electrode Holder and Optical Bench.*—The optical alignment and the source-to-slit distance shall be maintained accurately.

(*e*) *Photographic Processing Equipment,* providing developing, fixing, washing, and drying operations and conforming to the requirements of the Recommended Practices for Photographic Processing in Spectrochemical Analysis (ASTM Designation: E 115).[5]

(*f*) *Microphotometer,* capable of measuring the difference in transmittance of spectral lines with a standard deviation of 0.1 per cent transmittance.

(*g*) *Calculating Equipment,* for converting the microphotometer readings into per cent concentration. Such equipment shall have graphs or scales, or both, expanded sufficiently so that no significant error is introduced by calculations.

Reagents and Materials

4. (*a*) *Fusion Mixture.*—The reagents

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[6] The Patterson-Kelley 4-qt twin-shell blender has been found satisfactory for this purpose.

[7] A resistance furnace capable of maintaining 1150 C may also be used. Transformers, relays, pyrometer, and power controls are required to supply accurate temperature control. A special furnace type FHS-100, manufactured by Hoskins Manufacturing Co., is available for this application.

[8] R. C. Fowler and R. A. Wolfe, "A Non-Mechanical Interrupter for Controlled Spark Circuits," *Journal*, Optical Soc. Am., Vol. 35, pp. 170–174 (1945).

[9] The "Multisource," manufactured by Applied Research Laboratories, Inc., Glendale, Calif., has been found satisfactory for this purpose.

required for the fusion mixture are shown in Table I. These reagents (specified as purest grade fine powder) shall contain no constituent as an impurity that will interfere with the lowest concentration of that constituent in the sample. The dry components shall be weighed on an analytical balance or scale to an accuracy of ±0.01 per cent, and placed in a blender for thorough mixing. The uniformity of this mix determines to a major extent the accuracy and precision that can be obtained with this method. The mix may be prepared in the laboratory or it may be obtained commercially. Other fusion materials or mixes may also be satisfactory (Note 5).

TABLE I.—REAGENTS FOR FUSION MIXTURE.

Chemical	Form	Passing Sieve No.	Purpose	Per Cent by Weight
Sodium carbonate (Na_2CO_3)	dry powder	80 (177 μ)	flux	45.60
Sodium tetraborate ($Na_2B_4O_7 \cdot 5H_2O$)		80	flux	45.60
Columbium oxide (Cb_2O_5)		140 (105 μ)	internal standard	0.46
Nickel oxide (Ni_3O_4)		140	buffer	8.34

NOTE 5.—Zirconium oxide, vanadium pentoxide, or lithium carbonate can also be used as internal standards. For some materials it will be necessary to change the ratios of flux and buffer in order to effect a complete fusion.

(*b*) *Electrodes*.—Graphite rods[10] of commercial-grade purity or better shall be used for the supporting and counter electrodes. These shall be ¼-in. diameter rods cut to ½ ± 0.002-in. lengths. The tolerance on length is required to position accurately the analytical gap without touching the coated electrode end. The electrodes shall be uniformly flat on both ends and dust-free. Other lengths may be found convenient.

(*c*) *Photographic Emulsion*.—The spectrum shall be recorded on a high-contrast, fine-grain photographic plate or film that can be processed rapidly.

(*d*) *Photographic Processing Solutions*.—The formulas for processing solutions are given in the Recommended Practices E 115.

Standards

5. Primary or secondary standard samples for slag analysis are not available from commercial sources.[11] Secondary standards can be produced in the laboratory to cover the analytical concentration ranges for which it is necessary to calibrate. A series of such slags shall be prepared in the following manner: Select a slag sample of the approximate analysis to fulfill one requirement and prepare a convenient portion to pass a No. 100 (149-μ) sieve.[12] Mix thoroughly in a blender. Analyze portions of this sample carefully, preferably by more than one laboratory, and assign the average analysis as correct. Prepare additional standard samples in the same manner. Thoroughly blend mixtures of various proportions of high-composition and low-composition standards to provide a range of standards (Note 6).

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[10] These rods may be obtained in 12-in. lengths or cut to exact size from the National Carbon Co., New York, N. Y., or United Carbon Products Co., Bay City, Mich.

[11] "Report on Available Standard Samples and Related Materials for Spectrochemical Analysis," Am. Soc. Testing Mats. (1960). (Issued as separate publication, *ASTM STP No. 58-D*.)

[12] The sieves shall conform to the Specifications for Sieves for Testing Purposes (ASTM Designation: E 11), 1960 Supplement to Book of ASTM Standards, Parts 3 to 5 and 7 to 10.

NOTE 6.—Standard samples compounded synthetically with additions of metallic oxides are not always satisfactory. For some applications standards covering a wide range may be synthesized from two or three basic standards. As an example, by varying proportions of National Bureau of Standards samples No. 81 and No. 82, both silica standards containing 0.265 per cent and 1.96 per cent aluminum oxide (Al_2O_3) respectively, a complete curve covering the range between these values can be prepared.

Preparation of Standards and Samples

6. Crush the slag sample until it passes through a No. 100 (149-μ) sieve. Weigh 0.050 g of the sample or standard and 3.750 g of fusion mixture separately. Transfer the majority of the fusion mixture to a 25-ml platinum crucible and place the remainder on a clean glazed paper. Mix intimately the weighed slag with the fusion mixture on the glazed paper. Make an impression in the fusion mixture in the crucible and add the slag – fusion mixture from the glazed paper so that it does not touch the side of the crucible (Note 7). If difficulty is encountered with certain types of slag adhering to the crucible, place the sample directly on top of the fusion mix, and mix slightly by stirring with a platinum wire. Cover the crucible with a platinum lid, place in the load coil of a 2-kva induction furnace, and heat to 1150 C. When fusion is completed and the melt is quiescent, lower a piece of preheated $\frac{1}{4}$-in. diameter graphite rod into the melt until it just touches the bottom of the crucible. Stir the melt thoroughly and discard the rod. Preheat previously prepared $\frac{1}{4}$-in. diameter by $\frac{1}{2}$-in. long graphite electrodes, dust-free, to a low red heat by suspending them just above the melt before dipping. Immerse the electrode vertically in the melt to a depth of $\frac{1}{8}$ in., hold for about 5 sec, and then raise to a position just above the crucible until a cap of the melt solidifies. Place dipped electrodes (hygroscopic) in a storage block in an oven or under an infrared drying lamp at a minimum temperature of 105 C until used. Dip four electrodes in each melt (Note 8).

NOTE 7.—This technique is designed to prevent slag particles from sticking to the side of the crucible during fusion.

NOTE 8.—As many as ten or more electrodes can be prepared from a single melt. Standard samples may be prepared and stored for a month or more without deterioration if kept clean and dry.

Electrode System

7. Place the sample electrode in the lower electrode holder. Insert a graphite electrode in the upper electrode holder. Adjust to a gap of 3 mm *between the graphite surfaces*—not 3 mm between the fused surface of the sample electrode and the graphite surface of the counter electrode (Note 9). Because all the electrodes are the same length, this spacing is accomplished by gaging the gap from the back ends of the electrodes instead of from the ends forming the gap.

NOTE 9.—The gap may be varied to suit excitation conditions.

Excitation and Exposure

8. Produce and record spectra of the samples for analysis and standard samples for analytical calibration in exactly the same manner as follows:

(*a*) *Electrical Parameters* (Note 10):

Peak voltage, kv	15 to 35
Capacitance, μf	0.004
Inductance, μh	100
Radio-frequency current, amp	8.0 to 8.5
Discharge trains per half cycle, 60 cycle	10 to 12

NOTE 10.—These parameters are provided by an air-interrupted source. Other types of sources may also be employed provided proper parameters are used to produce the desired precision and accuracy.

(*b*) *Exposure Conditions:*

Spectral region, A	2500 to 3600
Slit width, μ	50
Preburn period	none
Spark exposure period, sec.	30 to 60
Neutral filter	transmittance of 38 per cent to cover half the slit length to provide two spectra of unequal transmittance from one exposure (Note 11).

NOTE 11.—Other means such as a stepped sector may be used to provide two spectra of unequal transmittance from one exposure.

(*c*) *Exposure Index.*—Adjust the time of exposure and the source-to-slit distance to provide a microphotometer reading on the unfiltered section of the Cb 2883.18 A line of approximately 65 per cent transmittance.

TABLE II.—ANALYTICAL LINE PAIRS.

Constituent	Analytical Line, A	Internal Standard Line, A	Concentration Range, per cent
CaO	Ca 3181.28	Cb 3191.10	15.0 to 40.0
	Ca 3179.33	Cb 3145.40	25.0 to 60.0
	Ca 3181.28	Cb 3145.40	35.0 to 58.0
SiO_2	Si 2881.58	Cb 2876.95	7.0 to 20.0
	Si 2881.58	Cb 2883.18	20.0 to 45.0
	Si 2524.12	Cb 2590.94	20.0 to 50.0
FeO	Fe 3020.64	Cb 3028.44	15.0 to 35.0
MgO	Mg 2790.79	Cb 3590.90	4.0 to 16.0
	Mg 2790.79	Cb 2883.18	4.0 to 25.0
Al_2O_3	Al 3092.71	Cb 3076.87	1.0 to 20.0
	Al 3082.16	Cb 3145.40	3.0 to 20.0
TiO_2	Ti 3088.02	Cb 3145.40	0.50 to 10.0
MnO	Mn 2933.06	Cb 2876.95	0.30 to 3.0
	Mn 2949.20	Cb 3145.40	1.00 to 5.0
	Mn 2933.06	Cb 2883.18	2.50 to 15.0

(*d*) *Replicate Exposure.*—Make duplicate exposures of all samples, preferably on separate plates or films.

Photographic Processing

9. Process the emulsion in accordance with Recommended Practices E 115.

Photometry

10. Set the microphotometer slit width so that the instrument measures two thirds of the width of the spectral line. Use a slit not less than 0.7 mm. Determine and record microphotometer readings of the series of iron lines selected for emulsion calibration. Determine the transmittance of the analytical line pairs appropriate to the mode of excitation and the concentration ranges listed in Table II (Note 12).

NOTE 12.—Other line pairs may also be satisfactory.

Calibration

11. (*a*) *Emulsion Calibration.*—Calibrate the emulsion in accordance with the Recommended Practices for Photometric Photometry in Spectrochemical Analysis (ASTM Designation: E 116).[5] Use the emulsion calibration curve to convert microphotometer readings for the element lines and internal standard lines to their log intensity ratios.

(*b*) *Preparation of Analytical Curves or Scales.*—Prepare the analytical curves or scales from data collected from a minimum of ten exposures each of standard slag samples. Follow the procedure as described in Sections 6 through 11. Establish the analytical curves by plotting the average intensity ratios against the respective element concentrations. Analytical scales may be produced from these curves if desired. Expose standard samples at sufficient intervals thereafter to assure that the calibrations are being maintained.

TABLE III.—DATA ON PRECISION OF METHOD.[a]

Element	Reported As	Average Concentration, per cent	Standard Deviation, per cent	Coefficient of Variation[b]
Calcium........	CaO	37.1	0.517	1.40
Silicon.........	SiO_2	15.6	0.348	2.23

[a] Data from 25 determinations of CaO and SiO_2 in one open-hearth slag sample.

[b] Coefficient of variation, ν, per cent is calculated as follows:

$$\nu = \frac{100}{\bar{X}} \sqrt{\frac{\Sigma\, d^2}{n-1}}$$

where:

$\bar{X}$ = average concentration, in per cent,

d = difference of the determination from the mean, and

n = number of determinations.

Calculations

12. Convert the microphotometer readings for the element lines and the internal standard lines to log intensity ratios. Refer these ratios to the appropriate analytical curves or scales to determine the per cent concentrations.

Precision and Accuracy

13. Data on precision of the method are given in Table III, and Table IV gives an example of the accuracy obtained by one laboratory for the analysis of open-hearth slags. Another laboratory reports the coefficient of variation for nearly all analyses of 1.0 to 1.5 per cent.

TABLE IV.—DATA ON ACCURACY OF METHOD.[a]

Constituent Reported as	Average Percentage by Spectrochemical Method	Average Percentage by Chemical Method	Standard Deviation Between Methods, per cent	Coefficient of Variation[b] Between Methods, per cent
CaO............	45.6	45.7	1.26	2.76
SiO_2............	16.8	16.0	0.77	4.70

[a] Data from 312 open-hearth slag samples.

[b] Coefficient of variation, ν, per cent, is calculated as follows:

$$\nu = \frac{100}{\bar{X}} \sqrt{\frac{\Sigma\, d^2}{n-1}}$$

where:

$\bar{X}$ = average concentration, in per cent,

d = difference of the determination from the mean, and

n = number of determinations.

DISCUSSION

This method has been used extensively in two laboratories. The techniques of fusing the sample and coating the electrode are not new. With modifications to the fusion mixture, this method has also proved useful for the analysis of silica brick, other refractories, fluxes, ores, and other nonconducting materials.

REPORT OF COMMITTEE E-3
ON
CHEMICAL ANALYSIS OF METALS*

Committee E-3 on Chemical Analysis of Metals held one meeting during the year: on June 29, 1960, in Atlantic City, N. J. in conjunction with the Annual Meeting of the Society. The Advisory Committee, divisions, and many task groups held individual sessions June 26–29, 1960.

The functions and activities within the committee are expanding rapidly and it has been necessary to add and re-align the divisional responsibilities as follows:

Division F on Ferrous Metals: Iron-base alloys, ferro-alloys, steel-making alloys, stainless and highly-alloyed steels, and heating alloys.
Division G on General Analytical Methods: Reagents, apparatus, statistical methods, procedures, and symposia.
Division M on Miscellaneous Metals: Basis alloys of chromium, hafnium, titanium, uranium, vanadium, zirconium, and metal powders.
Division N on Non-Ferrous Metals: Basis alloys of aluminum, beryllium, copper, indium, magnesium, tin, lead, zinc, alkali metals, semiconductor materials, precious or noble metals.
Division R on Refractory Metals: Basis alloys of columbium, molybdenum, rhenium, tantalum, and tungsten.
Division S on Sampling: Ferrous, non-ferrous, miscellaneous.

The International Organization for Standardization, on which the ASA is the U. S. representative, is assembling standard procedures for chemical analysis. The ASA is looking to Committee E-3 as a source to influence the ultimate quality of methods concerning metals.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the Annual Meeting, Committee E-3 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Methods for Chemical Analysis of:

Aluminum and Aluminum-Base Alloys (**E 34 - 60 T**), and
Special Brasses and Bronzes (**E 54 - 60 T**).

New Tentative Method of Test for:

Antimony in Copper and Copper-Base Alloys (**E 62 - 60 T**), and
Oxygen in Molybdenum (**E 174 - 60 T**).

New Tentative Recommended Practices for:

Conducting Inter-Laboratory Studies of Methods for Chemical Analysis of Metals (**E 173 - 60 T**).

Revision of Tentative Methods for Chemical Analysis of:

Ferroboron (**E 31 - 60 T**),
Titanium and Titanium-Base Alloys (**E 120 - 60 T**), and
Zirconium and Zirconium-Base Alloys (**E 146 - 59 T**).

Revision and Reversion to Tentative of Standard Methods for Chemical Analysis of:

Magnesium and Magnesium-Base Alloys (**E 35 - 60**), and
Silver Solders (**E 56 - 45**).

These recommendations were accepted by the Standards Committee on December 29, 1960, and the new and revised tentatives appear in the 1960 Book of ASTM Methods for Chemical Analysis of Metals.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

ACTIVITIES OF DIVISIONS AND SUBCOMMITTEES

Editorial Subcommittee (C. S. Mills, chairman) held three meetings during the year. A large number of new and revised methods were reviewed and several entire sections of older methods were editorially revised to conform to the presently-accepted form and style. Model methods have been selected to serve as guides for task forces.

Division G on General Analytical Methods (L. M. Melnick, chairman):

During the past year Methods E 50 (Apparatus and Reagents) and E 60 (Photometric Practices) were reviewed and brought up to date. Also, the Recommended Practices E 173 – 60 T were approved. A number of new and revised methods were reviewed prior to balloting.

A symposium on "Extension of Sensitivity for Determining Various Constituents in Metals" will be held at the June, 1961, meeting, in conjunction with Committee E-2 on Emission Spectroscopy.[1]

Division F on Ferrous Metals (J. J. Furey, chairman):

Projects on which Division F is currently active include the following:

Low-Carbon (less than 0.01 per cent) in Steel

A proposed preliminary method has been distributed to task force members. Three samples have been secured for evaluation of the method. Variability in accelerator, boats, and crucibles, and evaluation of Leco standards for preparation of calibration curves is being checked in four laboratories; two laboratories use resistance furnaces for combustion and two use induction type furnaces. When this work is completed, samples, boats, accelerators, and calibration standards will be distributed to all task force members for evaluation of the method.

Hydrogen In Steel

A detailed questionnaire was sent out to cooperators concerning operation of their equipment. As a result, stainless steel samples are being analyzed for hydrogen and when a sufficient number of appropriate samples have been obtained, they will be distributed to the task force for analysis. The upper limit of hydrogen concentration of interest has been set at 7 ppm and task force members will look for samples which will be tested for homogeneity in one laboratory before submission to other laboratories.

Low Sulfur (less than 0.01 per cent) in Steel

Samples for tests of both an umpire method and a combustion working method have been distributed to task force members. The umpire method is a procedure developed by Luke of Bell Telephone Laboratory. For the working method, a volumetric finish and a photometric finish will be tried out following combustion of the sample.

High Cobalt in Alloys

Cobalt sulfate has been adopted as a primary standard, following results obtained on a cobalt test metal. The number of results by the ion exchange method was insufficient to evaluate this procedure properly. When more results are in, the procedure will be tried by the entire task force.

Aluminum in Steel (Photometric)

A photometric procedure employing aluminon as a color reagent has been submitted for trial by the task force. A titration method applicable to high- and low-alloy steels will also be evaluated.

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[1] The papers in this symposium will be published as *ASTM STP No. 308*.

Nitrogen in Steel

The original method submitted for trial has not proved entirely satisfactory. The Armco Steel Co. method which is similar to the BISRA method will be tried.

Manganese in Ferromanganese and Silicomanganese

The task forces completed work on the modification of the Lingane-Karplus method for manganese in ferromanganese. The method was accepted and appears in the new edition of the book.

Ferrocolumbium

Methods for columbium, tantalum, titanium, silicon, carbon, manganese, aluminum, tin, and phosphorus have been sent out to task force members, together with a sample of the alloy. It is hoped to provide a National Bureau of Standards sample of ferrocolumbium as a corollary to the work of providing analytical methods for ASTM. The preliminary draft of the methods has been edited to conform to current ASTM style. Work on a photometric method for phosphorus in steels and on a photometric procedure for copper in steel employing neocuproine have been completed. The methods have been accepted and appear in the new edition of the book. A committee was appointed to investigate and report on the need for a method for boron in high-temperature alloys.

Tribute was paid to Messrs. Harry Bright and John Wiley, both of whom retired during the year, for their long service and worthwhile contributions to the Division.

Division N on Non-Ferrous Metals (R. G. Ernst, chairman):

The following new methods were completed and are included in the new edition of the book: (1) Method for the Determination of Zinc in Aluminum-Base Alloys with EDTA (Volumetric), (2) Method for the Determination of Zinc in Copper-Base Alloys with EDTA (Volumetric), and (3) Method for the Determination of Copper in Magnesium-Base Alloys with Neocuproine (Photometric). Also the entire group of procedures for the analysis of Magnesium and Magnesium-Base Alloys (Methods E 35) was revised editorially to conform to the most recent recommendations on form for methods of chemical analysis. Similar revisions of all of the groups of procedures under the jurisdiction of Division N are planned.

Work is still in progress on the following methods: (1) analysis of beryllium and beryllium oxide, (2) analysis of zinc anodes for cathodic protection, (3) determination of trace amounts of aluminum in zinc, (4) determination of minor constituents and impurities in silver brazing alloys, and (5) determination of beryllium in aluminum-base alloys (task force to be organized).

Agreement has been obtained with Subcommittee IX on Materials Analyses of Committee F-1 to attempt to combine the Methods for the Analysis of Electronic Nickel (E 107) with the Methods for the Analysis of Nickel (E 39). It is felt that the methods now recommended in E 107 can be improved by a less rigid restriction on sample size than had been imposed when these methods were first prepared.

Division M on Miscellaneous Metals (G. Porter, chairman):

Columbium Metal

Lack of satisfactory samples of boron has prevented any task force review. The conductometric method for carbon has proved satisfactory in the 400 to 600 ppm range and the review is being completed on tests with an 80 ppm sample. The data collected on hydrogen are being analyzed statistically and the method should be available shortly. A second task force review of iron is in progress. An improved dithiol method for molybdenum is being evaluated prior to a second round-robin program. Task force review of nitrogen is complete and the method is being balloted upon. Task force results on oxygen are encouraging, but lack of suitable range of metal for testing has prevented completion of the work. Preliminary evaluation of a method for silicon is taking place, prior to task force review. Very satisfactory results were obtained on an ion exchange method for tantalum. Preliminary arrangements have been made to supply the National Bureau of Standards with metal containing 15 important impurity elements.

Heating Alloys

The present methods are being reviewed prior to task force review on some new methods.

Lithium Metal

A task force chairman has been obtained and a program is being formulated.

Metal Powders

Group I task force review on hydrogen loss of iron powder was completed with satisfactory results and the method is now under editorial review. The Metal Powder Association method and supporting data for determining insoluble matter will be recommended for acceptance. A list of the slight modifications necessary to apply Committee E-3 methods to the analysis of metal powders has been submitted. In Group II task force, review of several methods for estimating the oxygen content of tungsten metal is continuing.

Molybdenum

Task force review of vacuum-fusion baths, other than the present iron-tin bath, for the determination of oxygen is dependent on obtaining samples of suitable metal. This group is also considering extending its scope to include tungsten metal.

Titanium

The volumetric method for aluminum is in the 1960 book. Two methods for carbon are undergoing task force review. A hot extraction method for hydrogen will be prepared. The tin and platinum flux methods for oxygen were approved during the year and are in the new book. The inert gas fusion method will be reviewed by task forces. The present methods for vanadium and chromium are being examined for possible weaknesses in the presence of certain alloying elements. The need for methods for zirconium and copper is being explored.

Zirconium Alloys

A method for tin had been proved in task force review, and is now in editorial review. Methods for carbon, tungsten, and uranium are being revised at task force level. Methods for oxygen and thorium are being tested.

Division S on Sampling (A. C. Holler, chairman):

Ferrous Subcommittee (J. B. Armstrong, chairman).—The task group on the sampling of high-carbon ferrous materials for the determination of the total carbon content has proposed a series of alternate methods of sampling. The materials that the proposed methods cover are cast iron, nodular iron, and pig iron. The results of this study are a part of Method E 59.

Division S is reviewing in cooperation with Committee E-2 on Emission Spectroscopy a method for the simultaneous sampling of molten ferrous alloys for routine control spectrographic and wet analysis.

Non-Ferrous Subcommittee (G. F. Donahue, chairman).—A sampling method for beryllium oxide has been circulated to Division S membership. The subcommittee is cooperating with Committee B-5 on Copper and Copper Alloys in a review of the Method of Sampling Wrought Non-Ferrous Metals and Alloys for Determination of Chemical Composition (E 55).

Miscellaneous Metals Subcommittee (John Danaczko, chairman).—Work is still in progress on the development of sampling methods for lithium, molybdenum, and columbium in cooperation with Subcommittee VIII of Committee B-2 on Non-Ferrous Alloys.

This report has been submitted to letter ballot of the committee, which consists of 120 members; 91 members returned their ballots, of whom 89 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

A. THOMAS,
Chairman.

H. KIRTCHIK,
Secretary.

REPORT OF COMMITTEE E-4
ON
METALLOGRAPHY*

Committee E-4 on Metallography, together with several of its subcommittees, met twice during the year: on June 26 through 28, 1960, during the Annual Meeting of the Society in Atlantic City, N. J., and on January 30 and 31, 1961, during ASTM Committee Week in Cincinnati, Ohio.

The committee presently consists of 124 members.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1960 Annual Meeting, Committee E-4 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method for:

Assigning Phase Designations in Metallic Systems (E 157 – 61 T).

Revision of Tentative Methods for:

Estimating the Average Grain Size of Metals (E 112 – 58 T).

Revision and Reversion to Tentative of Recommended Practice for:

Determining the Inclusion Content of Steel (E 45 – 51).

The new Tentative Method E 157 was accepted by the Standards Committee on January 3, 1961 and is available as a separate reprint. The revisions of Method E 112 and Recommended Practice E 45 were accepted on September 29, 1960 and they appear in the 1960 Supplement to Book of ASTM Standards, Part 3.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Methods for Estimating the Average Grain Size of Metals **E 112 – 60 T**)[1] be approved for reference to letter ballot of the Society for adoption as standard without revision.

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Methods for:

Preparation of Micrographs of Metals and Alloys (Including Recommended Practice for Photography as Applied to Metallography) (**E 2 – 49 T**), and

Determining the Orientation of a Metal Crystal (**E 82 – 49 T**).

Recommended Practice for:

Thermal Analysis of Metals and Alloys (**E 14 – 51 T**).

Definitions of Terms Relating to:

Metallography (**E 7 – 55 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] 1958 Book of ASTM Standards, Part 3.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Selection and Preparation of Samples (W. D. Forgeng, chairman) now has a number of task groups preparing recommendations on modern practices in preparation of metallographic samples in the several classes of metals and alloys. These will cover "micro" as well as "macro" samples.

Due to unforeseen and unavoidable circumstances, publication of the June 1960 symposium on polishing was materially delayed, but is now available as *STP No.* 285 on Methods of Metallographic Specimen Preparation.

Subcommittee III on Nomenclature (W. L. Fink, chairman), after many years of effort, has finally attained its goal of formal acceptance by the Society of the proposed Tentative Method for Assigning Phase Designations in Metallic Systems (**E 157 – 61 T**). Attaining universal use of the system is being materially aided by the adoption of the system for use in the ASTM Powder Data Card File. Additionally, an article has been published in *Materials Research & Standards*. Reprints of this article and of the system will be circulated nationally and internationally. Prior to the letter ballot, it had received widespread approval of eminent metallurgists at home and abroad. Contact will be maintained with all of the technical societies throughout the world that cooperated in the development of this method. It is hoped that these societies will help in the future improvements of the method and in securing universal use of the method.

A task group under the chairmanship of C. Schwartz has been appointed to devise an optional additional symbol that can be added to the phase designation to give extensive crystallographic information.

Another task group under the chairmanship of Taylor Lyman has been appointed to develop a standard method for presenting equilibrium data.

Subcommittee IV on Photography (G. A. Ellinger, chairman) is sponsoring the presentation of a paper describing a method of preparing macrographs of coarse-grained metals by means of point-source illumination.

The subcommittee is also revising the regulations for entries to the photomicrographic classes of the biennial Photographic Exhibit of the Society.

Subcommittee VI on X-ray Methods (R. K. Scott, chairman) is revising certain portions of Recommended Practice E 43. This is a temporary measure to up-date Recommended Practice E 43 pending the much enlarged version which will appear as a Special Technical Publication.

The subcommittee is also collaborating with the Joint Committee on Chemical Analysis by Powder Diffraction Methods in studying the possibilities of establishing a card index for electron diffraction patterns analogous to the well-known ASTM Powder Data Card File on X-ray patterns.

Subcommittee VIII on Grain Size (R. E. Penrod, chairman), completed Plate II, Twinned Grains-Flat Etch, for inclusion in the 1960 revision of Methods E 112. The subcommittee is firmly of the opinion that Methods E 112 will need no further changes in the foreseeable future, and thus recommends their adoption as standard.

Subcommittee IX on Inclusions (Samuel Epstein, chairman) revised the text of Recommended Practice E 45, and amplified the comparison chart in 1960.

Additional efforts are being made to include other methods of inclusion evaluation and also to extend these methods to application to vacuum-melted materials together with recommendations for quantitative use of the comparison chart.

Subcommittee XI on Electron Micro-

structure of Metals (C. M. Schwartz, chairman) is continuing its work on studies of alloys by the transmission electron microscopy of thin sections, as well as its studies on the structures of the super-strength, high-temperature alloys.

In addition, a new group has been formed to study electron diffraction methods. As a "methods" group, it will be closely coordinated with the electron diffraction data activities of Subcommittee VI.

Subcommittee XIV on Quantitative Metallography (G. E. Pellissier, chairman), although in the formative stage, has already accumulated several tasks in areas where reference standards are needed. These include such problems as evaluation of precipitates in stainless steels and measurements of carburization of steels.

This report has been submitted to letter ballot of the committee, which consists of 124 members; 99 members have returned their ballots, of whom 93 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. L. WYMAN,
Chairman.

MARY R. NORTON,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-4 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Recommended Practice for:

Determining Temperature-Electrical Resistance Characteristics (EMF) of Metallic Materials (E 189 – 61 T).

Withdrawal of Standard Methods for:

Estimating the Average Ferritic Grain Size of Low-Carbon Steels (E 89 – 52).

Withdrawal of Tentative Method for:

Estimating the Average Grain Size of Non-Ferrous Metals, Other Than Copper, and Their Alloys (E 91 – 51 T).

Withdrawal of Standard Classification of:

Austenite Grain Size in Steels (E 19 – 46).

Withdrawal of Tentative Recommended Practice for:

Identification of Crystalline Materials by the Hanawalt X-Ray Diffraction Method (E 43 – 49 T).

These recommendations were accepted by the Standards Committee on September 12, 1961, and the new tentative recommended practice appears in the 1961 Book of ASTM Standards, Part 3.

REPORT OF COMMITTEE E-5

ON

FIRE TESTS OF MATERIALS AND CONSTRUCTION*

Committee E-5 on Fire Tests of Materials and Construction held two meetings during the year: in Atlantic City, N. J. on June 29, 1960, and in Cincinnati, Ohio on February 3, 1961.

The committee consists of 75 members of whom 32 are classified as producers, 6 as consumers, 32 as general interest, and 5 as consulting members.

Revised by-laws of the committee were adopted during the year.

An Editorial Subcommittee was organized with the responsibility for reviewing all new and revised standards and tentatives.

The committee is proud to note that one of its members, S. H. Ingberg, was recognized by the Society at the 64th Annual Meeting as a 50-yr member, and the committee wishes to add its congratulations.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

On September 29, 1960, the Administrative Committee on Standards, acting for the Society, accepted the following recommendations submitted by Committee E-5:

New Tentative Methods of:

Test for Measuring the Surface Flammability of Materials Using a Radiant Heat Energy Source (E 162 – 60 T), and

Fire Tests of Window Assemblies (E 163 – 60 T).

Revision of Tentative Method of Test for:

Surface Burning Characteristics of Building Materials (E 84 – 59 T).

The new and revised tentatives appear in the 1960 Supplement to Book of ASTM Standards, Part 5.

NEW TENTATIVE

The committee recommends for publication as tentative the Definitions of Terms Relating to Fire Tests of Building Construction and Materials, as appended hereto.[1]

The first three definitions were submitted to the Administrative Committee on Standards on September 29, 1960, and were referred back to Committee E-5 for review of negative votes. Following this review, the definitions are submitted as part of this recommendation.

TENTATIVE REVISION OF STANDARD

The committee recommends the following tentative revision of the Standard Methods of Fire Tests of Building Construction and Materials (**E 119 – 58**):[2]

Section 9(b).—Change the first sentence to read as follows:

> Specimens are to be exposed to the controlled conditions outlined in Paragraph (*a*) until the interior or dampest section of the assembly attains a relative humidity of 70 per cent or less.

This recommendation is contingent on the recommendation set forth later in the report for adoption as standard of the tentative revision of Section 9 of the standard.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] The new tentative appears in the 1961 Book of ASTM Standards, Part 5.

[2] 1958 Book of ASTM Standards, Part 5.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Method of Test for Surface Burning Characteristics of Building Materials (**E 84 - 60 T**)[3] be approved for reference to letter ballot of the Society for adoption as standard without revision.

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee recommends that the tentative revisions[4] issued June, 1959, of Sections 9 and 30 of the Standard Methods of Fire Tests of Building Construction and Materials (**E 119 - 58**)[2] be approved for reference to letter ballot of the Society for adoption as standard.

REVISIONS OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as follows of the Standard Methods of Fire Tests of Building Construction and Materials (**E 119 - 58**),[2] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Section 4.—Change the title to read: "Temperatures of Unexposed Surfaces of Floors, Roofs, Walls, and Partitions." Change the first two sentences of Paragraph (*a*) to read as follows:

> Temperatures of unexposed surfaces shall be measured with thermocouples or thermometers[4] placed under flexible, dry, felted asbestos pads. The properties of these pads shall meet the requirements listed in Appendix II.

Add the following note to Paragraph (*a*):

> NOTE.—For the purpose of testing roof assemblies, the unexposed surface shall be defined as the surface exposed to ambient air.

Section 9(c).—Delete the last sentence.

New Appendix.—Add a new Appendix II as follows:

> *Appendix II.*—The asbestos pads used in measurements of temperature of unexposed surfaces of specimens shall be of felted amosite asbestos free of organic additives and shall exhibit the following properties:
>
> 1. Length and Width, 6 ± $\frac{1}{8}$ in.,
> 2. Thickness,[a] 0.40 ± 0.05 in.,
> 3. Dry Weight, 0.260 ± 10 per cent lb,
> 4. Thermal Conductivity (at 150 F), 0.38 ± 7 per cent Btu in. per hr sq ft deg Fahr, and
> 5. Hardness,[b] 10–25 (Modified Brinell).
>
> The pads shall be sufficiently soft so that, without breaking, they may be shaped to contact over the whole surface against which they are placed.

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[a] The thickness measurement is made under the light load of a ½-in. diameter pad of a dial micrometer gage.

[b] The hardness measurement is made by pressing a 1-in. diameter steel ball against the sample and measuring the indentation obtained between a minor load of 2 lb and an additional major load of 10 lb (12 lb total). The hardness is obtained by the relationship

$$\text{Hardness} = \frac{2.24}{y}$$

where y = the difference in indentation in inches.

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Method of Test for Defining Noncombustibility of Building Materials (**E 136 - 58 T**).

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for more than six years without revision:

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[3] 1960 Supplement to Book of ASTM Standards, Part 5.

[4] 1959 Supplement to Book of ASTM Standards, Part 5.

Standard Method of Test for:

Combustible Properties of Treated Wood by the Fire-Tube Apparatus (**E 69 – 50**), and
Combustible Properties of Treated Wood by the Crib Test (**E 160 – 50**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

SUBCOMMITTEE ACTIVITIES

Subcommittee I on Fire Tests of Materials and Construction (A. J. Steiner, chairman).—The work of a task group on development of new pads to replace existing ones has resulted in revisions of Section 4 and the addition of Appendix II to Method E 119 as indicated earlier in this report. Several additional changes were made in Method E 119 to improve the test procedure. Task groups are now actively studying end point criteria for ignition and also for beam deflection. A new task group was created to review the criteria for time of test as given in Section 9 of Method E 119.

Subcommittee IV on Fire Tests of Acoustical and Similar Finishes (C. H. Yuill, chairman) sponsored the 8-ft tunnel test which has been published as information, and the new tentative radiant panel test, referred to earlier, for research purposes. Several 8-ft and 25-ft tunnels are either now under construction or in operation. New correlation data have been distributed from the 25-ft tunnel apparatus from tests at the Underwriters' Laboratories, Inc. of Chicago and the Underwriters' Laboratories of Canada. A task group has been appointed to maintain and correlate information from various sources covering flame spread test performance and its relationship to actual structures.

Subcommittee V on Nomenclature and Definitions (J. V. Ryan, chairman) prepared the proposed tentative definitions for seven terms used in fire testing now in common usage, referred to earlier in the report. It is continuing work on additional definitions in this field.

Subcommittee X on Research (A. F. Robertson, chairman) sponsored a Symposium on Fire Test Methods at the Cincinnati meeting.[6] The subcommittee is now considering a symposium for some future meeting on the effects of moisture on the results of fire testing.

This report has been submitted to letter ballot of the committee, which consists of 70 voting members; 47 members returned their ballots, of whom 45 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

H. D. FOSTER,
Chairman.

I. A. BENJAMIN,
Secretary.

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[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[6] This has been published as ASTM Special Technical Publication *STP No. 301.*

REPORT OF COMMITTEE E-6

ON

METHODS OF TESTING BUILDING CONSTRUCTIONS*

Committee E-6 on Methods of Testing Building Constructions held one meeting during the year: on Nov. 14 and 15, 1960 in Washington, D. C., in conjunction with the fall meeting of the Building Research Inst. A highlight of this meeting was the Symposium on Testing which comprised five papers covering methods of testing various types of building assemblages. The co-chairmen of the symposium were R. F. Legget and D. E. Parsons.

The committee consists of 107 members of whom 78 are voting members; 48 are classified as producers, 5 as consumers, and 48 as general interest members, with 6 consulting members.

The committee paid tribute to Theodore Irving Coe, who died just prior to the November meeting. Mr. Coe's long and devoted service to technical groups, such as Committee E-6, will be long remembered.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as follows of the Standard Methods of Conducting Strength Tests of Panels for Building Construction (**E 72 - 55**)[1] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

New Method.—Add the following new Method *C* for Evaluation of Wetted Racking Panels, comprising new Sections 46 to 49, renumbering the present Sections 46 to 49 and subsequent sections accordingly:

Method C for Evaluation of Sheathing Materials (Wetted) on a Standard Wood Frame

This test has been developed to simulate the degree of wetting possible during construction of a structure when, because of rain, the framing and sheathing may be wetted on one or both sides. Both sides of the wall panel are wetted because this represents the maximum exposure possible during the stage of construction before the structure is roofed.

Test Specimens

46. The test specimens shall conform in size and fabrication details to the requirements of Section 43.

Conditioning

47. (*a*) The fabricated test specimens shall be mounted or suspended in a vertical position in such a manner as to prevent continuous immersion of the bottom edge of the specimen. Both sides of the test specimen shall be exposed to a water spray applied at or near the top along the entire length to insure that the top of the specimen is being wetted. The spray shall have no jet action that cuts

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] 1958 Book of ASTM Standards, Part 5.

into the sheathing material, and the spray areas shall overlap sufficiently so that a continuous sheet of water flows down both surfaces of the specimen. The temperature of the water in the line to the spray nozzle shall be maintained at 75 ± 5 F. The specimens shall be wetted for a period of 6 hr and then allowed to dry for a period of 18 hr. The drying shall take place in laboratory air, preferably at a temperature of 75 ± 5 F. No attempt shall be made to increase the air movement over the specimens by fans or blowers. The test specimens shall be subjected to two complete wetting and drying cycles and then a third wetting cycle. The racking test shall commence within 2 hr after the completion of the third wetting cycle. The racking test shall be carried out in accordance with Section 44.

(*b*) After the racking test is completed, moisture samples shall be cut from the sheathing material, and moisture content determinations shall be made on a weight basis with the moisture content expressed as a percentage of the oven-dry weight in accordance with Section 48(*b*). Preferably, five moisture content samples at least 4 by 6 in. in size shall be taken from each 4- by 8-ft sheathing panel of the test specimen; one from the center of each sheathing panel at the top and bottom edges, one from mid-length on each side, and one from the panel center.

Moisture Content

48. The moisture content samples shall be weighed immediately upon being cut from the test specimen to an accuracy of not less than ±0.2 per cent. All loose particles shall be carefully removed from the sample before weighing. The samples shall then be dried to constant weight in an oven at 103 ± 2 C (Note).

NOTE.—If large amounts of volatile matter or substances other than free water are removed from the sheathing material by drying at 103 ± 2 C, the sheathing material may be dried to constant weight at a lower temperature and the drying time and temperature given in the report.

Calculation

49. The moisture content shall be calculated as follows:

$$M = 100\left(\frac{W - F}{F}\right)$$

where:

M = moisture content, in per cent,
W = initial weight, and
F = final weight when oven dry.

Report

49. The report shall include the racking test data as specified in Section 45. It shall also include the line temperature of the water sprayed on the test specimens; the air temperature and relative humidity during the drying portion of the cycle; and the location of the moisture content samples and the moisture content of each.

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standard which has stood for more than six years without revision:

Standard Method of Testing:
Truss Assemblies (**E 73 – 52**).

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Method of Test for Bond Strength of Mortar to Masonry Units (**E 149 – 59 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

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[2] The letter ballot vote on these recommendations was favorable; the results of the ballot are on record at ASTM Headquarters.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Segments of Wall, Floor and Roof Construction (J. A. Liska, chairman).—A task group is preparing a clarification of Methods E 72 and replacing some of the illustrations with line drawings. The subcommittee made recommendations to the main committee regarding the development of performance criteria.

Subcommittee II on Durability (E. C. Shuman, chairman) is proceeding with the development of a recommended practice on durability of structures as affected by weathering with the scope to be limited to durability of a structure without considering individual materials.

Subcommittee III on Sound Transmission (R. K. Cook, chairman) is anticipating the completion of a revision of Recommended Practice E 90 by late spring 1961 and is considering the addition of information in an Appendix on classifying building elements.

Subcommittee VI on Completed Structures (W. R. Schriever, chairman) is balloting on loading tests for floors and flat roofs for publication as tentative.

Subcommittee VII on Vapor Barriers Beneath Concrete Slabs on the Ground (L. M. Wood, chairman) is accumulating data on the experience of users of E 154, Tentative Methods of Testing Materials for Use as Vapor Barriers under Concrete Slabs and as Ground Cover in Crawl Spaces.

Subcommittee VIII on Windows (L. M. Dunn, chairman) has established liaison with other ASTM committees having fields of mutual interest. A proposed standard on air leakage is being reviewed by the subcommittee.

Subcommittee IX on Curtain Walls (R. A. Biggs, chairman) held its first meeting on Nov. 15, 1960 and established four task forces on definitions, infiltration, strength, and durability.

This report has been submitted to letter ballot of the committee, which consists of 78 voting members; 63 members returned their ballots, all of whom voted affirmatively.

Respectfully submitted on behalf of the committee,

R. F. LEGGET,
Chairman.

J. P. THOMPSON,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-6 presented to the Society through the Administrative Committee on Standards the recommendation that the Recommended Practice for Laboratory Measurement of Airborne-Sound Transmission Loss of Building Floors and Walls (E 90 – 55) be revised and reverted to tentative. This recommendation was accepted by the Standards Committee on September 12, 1961, and the revised tentative recommended practice appears in the 1961 Book of ASTM Standards, Part 5.

REPORT OF COMMITTEE E-9
ON
FATIGUE

Committee E-9 on Fatigue held two meetings during the year: on June 28, 1960, at Atlantic City, N. J., and on Jan. 30, 1961, at Cincinnati, Ohio.

During the year, three members were elected to the main E-9 Committee. The committee lost one of its most active members in the passing of John M. Lessells who had been a member of Committee E-9 since its inception. He had played an important part in guidance of the committee and encouragement of its members until his death.

The officers elected at the June 1960 meeting for the ensuing term of two years are as follow:

Chairman, H. J. Grover.
Vice-Chairman, F. B. Stulen.
Secretary, R. F. Brodrick.
Assistant Secretary, S. M. Marco.

The committee gratefully acknowledges the many years of faithful leadership of R. E. Peterson, retiring chairman, and O. J. Horger, retiring secretary.

As in the past, the latter portion of each main meeting of Committee E-9 has been devoted to an informal presentation of a timely fatigue topic. Since these presentations are not published, the topics are recorded in this report: at the Atlantic City meeting, J. C. Grosskreutz of Midwest Research Inst. spoke on "The Mechanism of Fatigue Crack Propagation in Aluminum Single Crystals," and at the Cincinnati meeting, B. H. Schofield of Lessells and Associates, Inc. spoke on "Acoustic Emission Under Applied Stress."

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Research (G. M. Sinclair, chairman).—Upon the resignation of T. J. Dolan, the subcommittee was reconstituted under the chairmanship of G. M. Sinclair. The group will continue to carry on discussions and sponsor sessions on the continually expanding research activities in the field of fatigue.

Subcommittee II on Papers (D. G. Richards, chairman).—Papers have been reviewed for two sessions on fatigue at the 1961 Annual Meeting.

Subcommittee III on Survey (W. S. Hyler, chairman) is continuing the preparation of annual lists of references. In addition, progress is being made on the cross-indexing of all references listed in the STP No. 9 series. Consideration is being given to a new survey of current projects in fatigue.

Subcommittee IV on Apparatus and Test Methods (H. R. Neifert, chairman).—Following presentation of the Symposium on Large Machines, and the resignation of J. F. Millan, the Subcommittee on Large Machines and Test Correlation was terminated. A new Subcommittee IV, designated "Apparatus and Test Methods," was formed under the chairmanship of H. R. Neifert. Activities of the new subcommittee will encompass fatigue testing machines as well as detailed test procedures, including specimen preparation, etc.

Subcommittee V on Aircraft Structures (H. F. Hardrath, chairman) is preparing to form task groups on elevated temperature problems, low-cycle fatigue and residual static strength, full-scale tests, and helicopter problems. Sessions on low-cycle fatigue are being developed for presentation at the West Coast meeting in 1962. It is expected that three sessions will be held.

Subcommittee VI on Statistical Aspects of Fatigue (G. R. Gohn, chairman) held its organizational meeting in Cincinnati, Ohio. The major initial task concerns the proposed changes in STP No. 91-A, the Tentative Guide for Fatigue Testing and the Statistical Analysis of Fatigue Data. Questions regarding definitions will also be handled by this subcommittee.

This report has been submitted to letter ballot of the committee, which consists of 58 members; 45 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. J. GROVER,
Chairman.

R. F. BRODRICK,
Secretary.

REPORT OF COMMITTEE E-10

ON

RADIOISOTOPES AND RADIATION EFFECTS*

Committee E-10 on Radioisotopes and Radiation Effects held two meetings during the year: on June 30, 1960, in Atlantic City, N. J., and on Feb. 1, 1961, in Cincinnati, Ohio.

The committee consists of 137 ASTM members plus 63 consultants and appointed task force specialists.

Robert S. Shane was appointed chairman of a Space Radiation Study Group. This group has met three times during the year to determine the proper course of Committee E-10 regarding activities in the space-radiation field. This group gave a report on this subject at the 1961 Committee Week Meeting in Cincinnati. As a result of the interest stimulated, Committee E-10 will sponsor a panel discussion on "Space Radiation and Its Effects on Materials" at the 1961 Annual Meeting in Atlantic City.

Subcommittee I on Health and Safety was abolished and replaced by Subcommittee I on Education and Information, whose scope and objectives are: publicity and education, establishment of a speakers bureau, and liaison with ASTM and related other groups.

Subcommittee V on Dosimetry was formed with James B. Trice as chairman. The scope of this subcommittee is as follows:

> The Subcommittee on Dosimetry shall provide advice and counsel to other ASTM committees concerning all matters of dosimetry of materials that are subject to ionizing radiation from any source. It will also write ASTM standard methods of dosimetry to encompass all exposures to ionizing radiation.

J. V. Alger was appointed chairman of Subcommittee II on Radiation-Induced Changes in Materials. R. R. Quinn was appointed Assistant Secretary of Committee E-10.

Membership on the Advisory Subcommittee was redefined and limited to committee officers, past chairmen, and subcommittee chairmen. Scopes and memberships of each committee were evaluated and a committee manual is now being prepared.

The committee regretted receiving the resignation of George C. Manov because of his change of professional affiliation. Mr. Manov was instrumental in establishing Committee E-10 and was its first chairman.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods and recommended practices as appended hereto:

Tentative Methods for:

General Methods for Analysis of Radioisotopes,[1] and

Analysis of Phosphorus-32.[1]

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] The new tentative methods appear in the 1961 Book of ASTM Standards, Parts 3, 7 and 10.

Tentative Recommended Practices for:

Determining Changes in the Chemical Reactivity of Inorganic Materials Exposed to High Energy Radiation,[2]

Effect of High-Energy Radiation on the Tensile and Impact Properties of Metallic Materials,[3] and

Surveillance Tests on Structural Materials in Nuclear Reactors.[3]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Radiation Induced Changes in Material (J. V. Alger, chairman) has had several task force meetings during the year and will sponsor a symposium on "Radiation Effects in Refractory Fuel Compounds"[5] at the June, 1961, Annual Meeting. This symposium will be followed by another at the October, 1962, ASTM Meeting in Los Angeles on "Non-Fuel Structural Materials." This subcommittee is preparing several tentatives, three of which are currently out for ballot.

Subcommittee III on Tracer Applications (John L. Kuranz, chairman) has prepared two tentatives which are out for ballot and is currently preparing others. Three of its earlier proposed tentatives were withdrawn pending clarification with other ASTM committees. W. S. Lyons has been appointed vice-chairman.

Subcommittee V on Dosimetry (James B. Trice, chairman) currently has three tentatives out for ballot on cesium, nickel, and measuring neutron-flux environments which are expected to be ready for Committee E-10 ballot vote sometime during this year. The subcommittee currently has several other tentatives under development.

This report has been submitted to letter ballot of the committee, which consists of 137 members; 71 members have returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

E. B. ASHCRAFT,
Chairman.

O. KENTON NEVILLE,
Secretary.

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[2] The new tentative recommended practice appears in the 1961 Book of ASTM Standards, Parts 3 and 4.

[3] The new tentative recommended practices appear in the 1961 Book of ASTM Standards, Part 3.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[5] Published as *ASTM STP No. 306*.

REPORT OF COMMITTEE E-11

ON

QUALITY CONTROL OF MATERIALS*

Committee E-11 on Quality Control of Materials held one meeting during the year: on November 10, 1960, at Society Headquarters in Philadelphia. The Advisory Committee met on November 9, 1960 in Philadelphia, and on February 23, 1961 in New York City.

The committee's activities, other than the preparation of the standards referred to below, have been concerned with the preparation of a manual on fitting straight lines which is being recommended for publication as a Special Technical Publication. Work continues on the development of a manual on planning and conducting interlaboratory test programs. Consideration is being given to a possible reorganization of the committee structure so as to deal more effectively with the problems of the Society in the field of applied statistics.

A new task group to handle editorial reviews of documents that have been approved for publication has been organized with Paul S. Olmstead as chairman.

NEW TENTATIVES

The committee recommends for publication as tentative the following recommended practices as appended hereto:[1]

Tentative Recommended Practices for:

Use of the Terms Precision and Accuracy as Applied to Measurement of a Property of Material, and

Dealing with Outlying Observations.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Recommended Practice for the Acceptance of Evidence Based on the Results of Probability Sampling (**E 141 – 59 T**)[2] be approved for reference to letter ballot of the Society for adoption as standard without revision.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

This report has been submitted to letter ballot of the committee which consists of 26 members; 26 members returned their ballots, of whom 24 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

SIMON COLLIER,
Chairman.

D. H. W. ALLAN,
Secretary.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] The new tentatives appear in the 1961 Book of ASTM Standards, Parts 3, 4, 5, 6, 7, 8, 9, 10, and 11.

[2] 1959 Supplement to Book of ASTM Standards, Parts 3 to 10.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE E-12
ON
APPEARANCE*

Committee E-12 on Appearance met twice in the past year: at Atlantic City, N. J., on June 30 during the Annual Meeting of the Society and again at Cincinnati, Ohio, on February 2 and 3, 1961 during ASTM Committee Week.

In contrast to the programs in previous years of symposia on selected subjects in this field, the past 12 months were devoted to work on a variety of projects. These will later be prepared for recognition via symposium or publication.

Task Group 1 on Manual on Appearance (I. Nimeroff, chairman) has made substantial progress towards its objective of preparing a manual concerned with the aims, procedures, terminology, and uses of appearance measurements. After several years of shaping this project, enough of its form and content is sufficiently evident to have already attracted inquiry from other technical societies with related interest.

Task Group 4 on Standards for Aluminum Finishes continues its research in preparing standard procedures for measurement of color and gloss of variously treated and unfinished aluminum surfaces. This was recognized as a "Herculean Task" by editors of the English publication *Metal Industry.* A number of publications will result from this work and one has been selected for presentation at a technical symposium in England next September. Recognized by this group for authoritative treatment of the fundamentals involved in this work, D. B. Judd's paper on "A Five Attribute System of Describing Visual Appearance" was revised by the author for publication.[1]

Task Group 5 on Typewriter Ribbon and Carbon Paper (W. E. Grady, chairman) continues its extensive efforts to measure and standardize the performance of typewriter ribbon and carbon paper and is solving a variety of stubborn, long-standing research problems. Experts in various fields of appearance-test methodology have been attracted to this project. This cooperative program is an excellent example of what can be accomplished under Committee E-12 sponsorship.

New approaches are being taken to identify problem areas to improve methods of pictorial representation in standards as well as in technical publications, of all types.

Subcommittee III on Pictorial Representation (J. M. Hemphill, chairman) is recruiting personnel with demonstrated talent, primarily industrial photographers adept at morphological analyses. These individuals should contribute new ideas in representing appearance properties.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] This paper has been published as *ASTM STP 297.*

The services of a representative of the technical literature field, R. J. Fabian, will serve as chairman of Subcommittee I of Intercommittee Relations. He will vitalize the relations with technical committees by identifying their problems which are of real industrial significance. His effort should generate appropriate publicity within and outside the society to bring to our attention problems worth solving, and solutions worth publicizing.

NEW TENTATIVE

The committee recommends for publication as tentative the Recommended Practice for Selection of Geometric Conditions for Measurement of Reflectance and Transmittance as appended hereto.[2]

STANDARDS CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Standard Method of Test for 45-deg, 0-deg Directional Reflectance of Opaque Specimens by Filter Photometry (**E 97** – 55).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Recommended Practice for:

Goniophotometry of Transmitting Objects and Materials (**E 166 – 60 T**), and
Goniophotometry of Reflecting Objects and Materials (**E 167 – 60 T**).

This report has been submitted to letter ballot of the committee, which consists of 60 members; 55 members returned their ballots, of whom 50 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

G. W. INGLE,
Chairman.

S. H. PETRY,
Secretary.

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[2] The new tentative appears in the 1961 Book of ASTM Standards, Parts 6, 8, and 9.

REPORT OF COMMITTEE E-13
ON
ABSORPTION SPECTROSCOPY*

Committee E-13 on Absorption Spectroscopy and most of its subcommittees met twice during the year: in New York City on Nov. 2 and 3, 1960, in conjunction with the Eastern Analytical Symposium, and on Feb. 26 and 27, 1961, with the Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy.

A summary of the activities, accomplishments, and unsolved problems of the committee and its subcommittees was published during the year.[1]

Committee E-13 plans to meet again with the Eastern Analytical Symposium in New York in November, 1961, and with the Pittsburgh Conference in March, 1962.

New Title and Scope.—The committee plans to recommend to the Board of Directors the following new title and scope to represent better the field of activity. The field includes some areas not strictly absorption spectroscopy, such as molecular fluorescence. The presentation of these changes will await the completion of corresponding changes in the title and scope of Committee E-2 on Emission Spectroscopy.

Title.—Committee E-13 on Molecular Spectroscopy.

Scope.—The advancement of the field of molecular spectroscopy involving absorption, fluorescence, scattering, or polarization of radiant energy, by promoting exchange of information, by sponsoring meetings and symposia for presentation of papers, by standardizing nomenclature relating to molecular spectroscopy, and by coordinating and formulating scientific practices and methods of analysis.

PROPOSED METHOD TO BE PUBLISHED AS INFORMATION

The committee recommends for publication as information only the Proposed Recommended Practices for Identification of Materials by Absorption Spectroscopy Using the Wyandotte–ASTM (Kuentzel) Punched Card Index, as appended hereto.[2] This method, recommended to the committee by Subcommittee II on Methods, describes the use of the Wyandotte - ASTM (Kuentzel) Punched-Card Index of Absorption Spectral Data[3] maintained by Subcommittee III on Standard Data.

REVISION OF TENTATIVE

The committee recommends revision as follows of the Tentative Definitions of Terms and Symbols Relating to Absorption Spectroscopy (**E 131 - 59 T**)[4] and continuation of the definitions as tentative. The revisions were prepared by Subcommittee IV on Nomenclature.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] R. F. Robey, "Ten Years of Aid to Applied Absorption Spectroscopy," *Applied Spectroscopy*, August, 1960, pp. 103–106.

[2] See p. 653.

[3] The Card Index may be purchased from ASTM Headquarters.

[4] 1959 Supplement to Book of ASTM Standards, Part 7.

New Definitions.—Add the following new definitions in their proper alphabetical sequence:

Baseline.—Any line drawn on an absorption spectrum to establish a reference point representing a function of the radiant power incident on a sample at a given wavelength.

Derivative Absorption Spectrum.—A plot of rate of change of absorbance or of any function of absorbance with respect to wavelength or any function of wavelength, against wavelength or any function of wavelength.

Difference Absorption Spectrum.—A plot of the difference between two absorbances or between any function of two absorbances, against wavelength or any function of wavelength.

Radiant Energy.—Energy transmitted as electromagnetic waves.

Spectral Band Width.—The wavelength or frequency interval of the radiation leaving the exit slit of a monochromator between limits set at a radiant power level half way between the continuous background and the peak of an emission line or an absorption band of negligible intrinsic width.

This report has been submitted to letter ballot of the committee, which consists of 89 members; 72 members returned their ballots, of whom 60 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

E. J. ROSENBAUM,
Chairman.

R. F. ROBEY,
Secretary.

APPENDIX

PROPOSED RECOMMENDED PRACTICES FOR IDENTIFICATION OF MATERIALS BY ABSORPTION SPECTROSCOPY, USING THE WYANDOTTE-ASTM (KUENTZEL) PUNCHED CARD INDEX[1,2]

This is a proposed recommended practice and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. (*a*) These recommended practices describe procedures for identification of individual chemical substances using infrared (including near-infrared and far-infrared), visible, or ultraviolet absorption spectroscopy, and card indexes of spectral data. Use of absorption spectroscopy for qualitative analysis has been described by many (**1** to **5**),[3] but the rapid matching of the spectrogram of a sample with spectral data in the literature by use of a card index system designed for machine sorting was contributed by Kuentzel (**6**). It is on Kuentzel's system that the Wyandotte - ASTM indexes of absorption spectral data are based.

(*b*) Use of these recommended practices requires, in addition to a recording spectrometer and access to published spectral data, an appropriate deck of the Wyandotte - ASTM punched cards[4] and a suitable sorter.[5]

Summary of Method

2. A representative sample of the material to be analyzed is separated into its individual components, if required, and each component is introduced into a suitable sample cell or matrix, mainly according to its physical state. The spectrogram is recorded with a double-beam infrared or ultraviolet spectrophotometer over a conventional range of the spectrum. The first choice of spectral range and instrument is dictated by a general consideration of the chemical nature of the sample (**3**). A note is made of the spectral positions of prominent absorption bands and of any previous knowledge of the chemical and physical properties of the material. The qualitative chemical composition of the material may then be determined by machine sorting of the appropriate decks of a coded card index[4] to isolate the card or cards for the chemical compound or compounds having matching characteristics. Details on preparation of suitable spectrograms and on the card index, codes, and sorting procedures are available in a book of codes and instructions.[4]

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[1] This proposed recommended practice is under the jurisdiction of the ASTM Committee E-13 on Absorption Spectroscopy.

[2] Published as information only, June, 1961.

[3] The boldface numbers in parentheses refer to the list of references appended to these recommended practices.

[4] The Wyandotte - ASTM Punched-Card Index of Spectral Absorption Data may be purchased as a whole, or by section or spectral region, from the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa. The Society furnishes purchase information upon request. A "Book of Codes and Instructions for Wyandotte - ASTM Punched Cards Indexing Spectral Absorption Data" is furnished without charge to purchasers of the index cards. The book of codes and instructions and unpunched index cards are available separately at nominal prices.

[5] Sorting operations require use of a standard Type 82, 80-1, 80-2, or 101 sorters. Sorters are available on a rental or purchase basis from International Business Machines, Inc. (IBM).

Apparatus

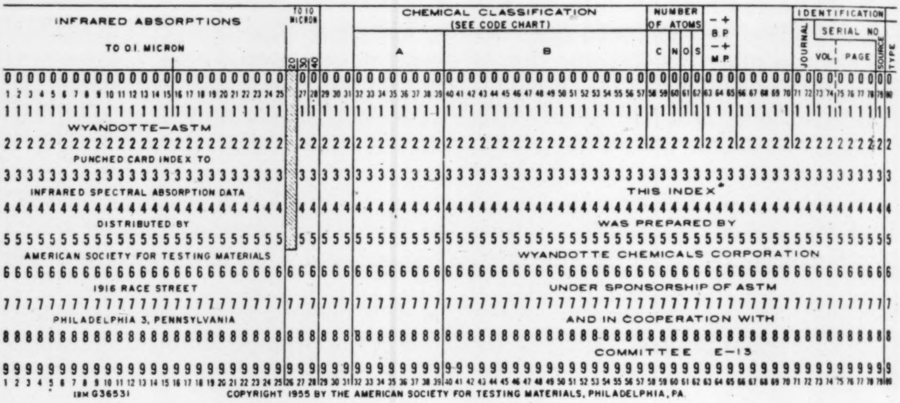
3. One, and sometimes both, of the following spectrophotometers will be required, depending upon the chemical nature of the sample:

(*a*) *Infrared Spectrophotometer*.—A recording double-beam instrument is much to be preferred, operating with about "Condition C" spectral resolution and 0.05-μ wavelength accuracy over the spectral range 2 to 15 μ as determined in accordance with the Proposed Methods for Evaluation of Spectrophotometers.[6] The range up to 36 μ (far-infrared) can be attained by modification of the ordinary instrument or by special instruments offered by the manufacturers.

(*b*) *Ultraviolet Spectrophotometer*.—A recording double-beam instrument is much preferred, operating with about "Condition C" spectral resolution and 1 to 5-mμ wavelength accuracy over the spectral range 200 to 2700 mμ, as determined in accordance with the Proposed Methods for Evaluation of Spectrophotometers.[6] This spectral range covers the ultraviolet, visible, and near-infrared regions. Recording spectrophotometers that operate only in ultraviolet and visible ranges, or exclusively in the visible range, and meet the above requirements, are limited in applicability but quite permissible.

(*c*) *Spectrophotometric Accessories*.—The type of accessories needed depends upon the physical state of the sample and the technique used as described in Section 5.

(*d*) *Card Sorter*.[5]


INFRARED ABSORPTIONS
TO 0.1 MICRON
TO 10 MICRON
20 30 40
CHEMICAL CLASSIFICATION (SEE CODE CHART)
A
B
NUMBER OF ATOMS
C N O S
−+ B.P −+ M.P
IDENTIFICATION
JOURNAL
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TYPE
WYANDOTTE—ASTM
PUNCHED CARD INDEX TO
INFRARED SPECTRAL ABSORPTION DATA
DISTRIBUTED BY
AMERICAN SOCIETY FOR TESTING MATERIALS
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AND IN COOPERATION WITH
COMMITTEE E-13
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FIG. 1.—IBM Punched Card for Wyandotte - ASTM Index to Infrared Spectral Absorption Data

Card Index

4. (*a*) Although IBM (International Business Machine) cards and sorters are quite commonplace, the spectroscopist may be faced with the necessity of becoming more familiar with such tools in order to make use of the many advantages. The techniques are simple and the equipment inexpensive when one considers the results that can be achieved.

(*b*) An IBM card is 3.25 by 7.375 in. in size and, as shown in Fig. 1, contains eighty vertical columns of numbers from 0 through 9. These numbers mark the positions where small rectangular holes are punched into the cards to record coded data. In card sorting operations, the simplest IBM sorters operate upon one column at a time. As the cards pass over a metal cylinder, a small metal brush makes electrical contact with the cylinder through the holes in the cards. This causes the card to be shunted into a pocket corresponding to the numbered position in the card at which a punch occurs. Thus the machine isolates all cards having the same code punches. Fig. 2 is a photograph of an IBM card-sorting machine in operation on a deck of punched cards indexing spectral absorption data. This operation may be attended by a laboratory technician or an IBM-machine operator, preferably under supervision of a spectroscopist.

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[6] *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 472 (1958).

(*c*) The card index[4] comprises decks of different cards because the character of the data and some of the codes vary somewhat according to the spectral region. The spectral regions covered by individual decks of cards are as given in Table I. The decks are designed to cover all suitable spectral data, and supplementary and revised cards are scheduled[7] for issuance as fast as data are published. Included in the present index are cards

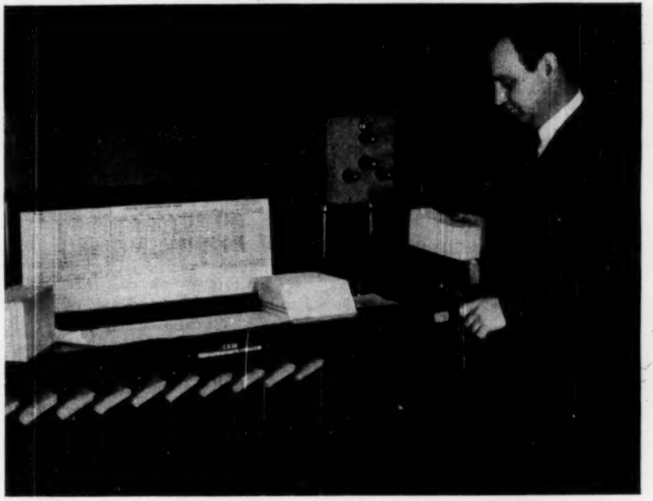

FIG. 2.—Matching of Spectral Data of Unknown Substance by Machine Sorting of Spectral Index Cards.

TABLE I.—SPECTRAL REGIONS COVERED BY INDEX CARDS.

Spectral Data Index Deck	Spectral Range	Approximate Number of Cards in Deck[a]
Far-infrared	11 to 36 μ	450
Regular infrared	2 to 16 μ	34 000
Near-infrared	0.7 to 3.4 μ (700 to 3400 mμ)	1 800
Visible	350 to 845 mμ	1 300
Ultraviolet	200 to 420 mμ	13 000

[a] As of February, 1961.

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[7] Gathering and coding of spectral data from the literature and from readily available organizational and commercial files is the responsibility of the Subcommittee on Standard Data of the ASTM Committee E-13 on Absorption Spectroscopy. Card punching and distribution are administered by the ASTM Headquarters.

for such collections of spectra as are indicated in Table II for the infrared range. In all ranges, the card index represents a very good coverage of available spectral data. Although it does not cover every chemical compound that might be encountered, use of the index in the manner described will sometimes point to closely related chemical substances. Spectral and other data obtained on chemical substances in the user's own laboratory may be coded into the system by punching the data into the index or extra cards.

In addition to the spectral data of individual chemical substances coded into these cards, there are common codes for chemical structure classification, empirical formula, melting or boiling points, and serial number references. Other codes, which vary, include data on solvents, spectral peak intensities, and numbers of peaks. For a given chemical substance the pattern of holes in the card, corresponding to coded spectral data, is almost invariably unique as is the pattern for coded chemical structure and physical properties. All cards may be searched by machine in any desired combination (*1*) to locate a standard spectrum similar to that of a sample of unknown composition, (*2*) to correlate type of structure with absorption band positions, (*3*) to locate spectra of compounds having given structural features in common, and (*4*) in many other ways that are too numerous to include here but become evident upon study of the codes and instructions.

(*d*) Empirical formula - name cards comprise other decks for use in conjunction with those listed above. These cards carry the empirical formulas, chemical names, and reference serial numbers for the compounds included in the card indexes as described in Paragraph (*c*). This information is also printed across the top of the card. The cards do not require a sorting machine and their purpose is largely twofold: (*1*) When arranged in numerical order of the serial number the cards provide direct correlation between spectrum serial number and name of compound, and (*2*) when arranged in numerical order of the empirical formulas the cards provide direct correlation between name or formula of a compound and its published spectrum. Thus, to identify a compound it is not always necessary to procure all the catalogs of spectrograms (for example, those listed

TABLE II.—CATALOGS OF SPECTROGRAMS COVERED BY WYANDOTTE-ASTM PUNCHED CARDS INDEXING INFRARED ABSORPTION DATA.

A. API Research Project 44[a]
B. User's own file of spectrograms[b]
C. Sadtler catalog of spectrograms[c]
D. NRC-NBS file of spectrograms[d]
E. Literature[e]
F. Documentation of Molecular Spectroscopy[f]
G. Coblentz Society Spectrograms[c]
H. Manufacturing Chemists Association[g]
J. Infrared Data Committee of Japan[h]

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[a] American Petroleum Institute, Research Project 44, "Infrared and Ultraviolet Spectral Data," Carnegie Institute of Technology, Chemical and Petroleum Research Laboratory, Pittsburgh 13, Pa., 1943 to date. Looseleaf.

[b] Users are encouraged to submit spectrograms (or the pure compound in some cases) to one of the other organizations listed. It is unlikely that any individual laboratory can code its spectral data and punch cards at the cost of the Wyandotte-ASTM cards (about one cent each).

[c] The Sadtler Research Laboratories, "Standard and Commercial Spectra," Philadelphia 2, Pa. Looseleaf. The Sadtler organization also offers a "Spec-Finder" book method of matching spectrograms with those in its catalog.

[d] National Research Council-NBS Committee on "Spectral Absorption Data," National Bureau of Standards, Washington 25, D.C. Card file.

[e] Numerical list furnished without charge by ASTM Headquarters to subscribers of the card Index.

[f] "The DMS System," Butterworth Scientific Publications, London WC2. Distributed in U. S. by Spex Industries, Inc., Scotch Plains, N. J. Card file.

[g] Manufacturing Chemists Association, Inc., 1825 Connecticut Ave., N. W., Washington 9, D. C. Looseleaf.

[h] Infrared Data Committee of Japan, Sanyo Shuppan Doeki Co., Inc., Hoyu Bldg., 8, 2-chome, Takara-cho, Chuo-ku, Tokyo, Japan. Card file.

in Table II); however, it is desirable to have as many as feasible on hand for detailed comparison and study, because positive identification depends upon matching the spectrum with one from published material or with one obtained on a bona fide sample of the compound. A definite convenience is offered by use of numerical and alphabetical lists of the spectrograms in available catalogs. Most publishers of catalogs of spectra supply, at a nominal price, numerical lists in printed form that readily serve the same purpose as the numerically arranged formula-name cards and some provide alphabetical or formula lists which assist in the purpose of the alphabetically arranged formula-name cards, but the formula-name cards are a most complete, single index to the published spectra.

(*e*) The book of codes and instructions[4] that accompanies the card index describes the codes used in indexing absorption data in each spectral range and the details of sorting techniques.

Sample Preparation

5. (*a*) These recommended practices are most suitable for use with single chemical compounds and simple mixtures. Complex mixtures should usually be separated into individual components by extraction, distillation, chromatography, or other well-known techniques prior to the use of infrared or ultraviolet spectrophotometry to identify the individual components. Recommended practices in infrared and ultraviolet spectrophotometry are covered in the Recommended Practices for General Techniques of Infrared Quantitative Analysis (ASTM Designation: E 168)[8] and the Recommended Practices for General Techniques of Ultraviolet Quantitative Analysis (ASTM Designation: E 169).[8]

(*b*) *Infrared Spectrophotometric Technique:*

(*1*) Gaseous material most commonly requires a 5 or 10-cm sample cell equipped with standard sodium chloride (NaCl) window material. A vacuum pump and gas-handling system for drying the sample and for introducing it into the sample cell at a known pressure are also required.

(*2*) Liquids are examined most often undiluted in a fixed-thickness cell. Thickness of 0.05 to 0.1 mm is recommended for nonpolar materials such as aliphatic hydrocarbons and 0.01 to 0.025 mm for materials containing aromaticity, unsaturation, oxygen, nitrogen, halogen, etc. A variable thickness cell is ideal for qualitative work. The common cell-window material is again sodium chloride; potassium bromide (KBr) is most often used for long wavelength work to 25 μ.

(*3*) Most solids are amenable to the following potassium bromide pellet technique (**7,8**). One part of the sample is ground with 200 parts by weight of spectrophotometric grade potassium bromide in an atmosphere of dry nitrogen. The finely powdered mixture is inserted in a special die and pelletized with a hydraulic press. The pressure produces a transparent or translucent disk that is supported in an available accessory. The optimum thickness of the disk for the above mixture is 0.5 mm as measured by a micrometer. Other techniques of preparing solids include mulling in USP grade mineral oil[9] or perfluorocarbon oil, melted or pressed film, and solution in carbon disulfide and symmetrical carbon halides. These techniques and the use of spectrophotometric accessories are described in the literature of the instrument manufacturer.[10]

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[8] 1960 Supplement to Book of ASTM Standards, Part 7.

[9] Nujol brand mineral oil has been found satisfactory for this purpose.

[10] An example of such literature is the "Instruction Manual, Vol. 2, Infrared Sampling and Techniques," The Perkin-Elmer Corp., Norwalk, Conn. Other instrument manufacturers furnish equivalent manuals.

(*c*) *Ultraviolet Spectrophotometric Technique:*

(*1*) Most work in the ultraviolet and visible ranges can be done with a matched pair of silica cells having 1.0-cm optical path. One cell contains a solution of the sample, most often with spectrophotometric grade *iso*octane or alcohol as solvent, depending upon the solubility of the sample. The other cell contains the pure solvent as reference. It is suggested that 10 ml of an initial solution should contain 0.1 g of sample with subsequent solutions being made by successive tenfold dilutions to the extent required to attain optimum absorbance as described in Section 7.

(*2*) In the near-infrared spectral range, liquid samples are often examined undiluted in cells of 10, 1, and 0.1-cm optical paths. The cell windows are quartz. Matching cells may also be used for solutions containing an appropriate concentration of sample in spectrophotometric grade carbon tetrachloride (**Caution,** Note 1) with the pure carbon tetrachloride as the reference material.

NOTE 1: **Caution.**—Vapors of carbon tetrachloride are toxic; use hood for sample preparation.

Procedure for Infrared, Near-Infrared, and Far-Infrared Technique

6. (*a*) Record the spectrogram of the material in such a thickness and concentration that the peak of the strongest absorption band lies between 1 and 20 per cent transmittance or the corresponding absorbance, if at all feasible.

(*b*) Note the spectral positions of the prominent absorption bands to the nearest 0.1 μ for the infrared and far-infrared, and 0.01 μ (100 mμ) for the near-infrared, as shown in the book of codes and instructions.[4] Recall any other characteristics of the material, such as elemental composition, an unusual functional group or physical property, that are known without doubt and are covered by the codes in the card index.

(*c*) Allowing for errors of 0.1 and 0.01 μ in the location of infrared and near-infrared bands, or a spread of 0.3 and 0.03 μ, respectively, sort the appropriate deck of index cards according to the directions in the book of codes and instructions.

(*d*) Follow instructions in the manual with respect to "negative sorting," sorting systematically by working from the long wavelength region toward the short or from the known characteristics of the compound, "presorting," etc. (See the supplementary discussion in the Appendix.) This will speed the isolation of the card or cards corresponding to the compound or compounds, if such a card exists in the index deck. Note the reference serial number or numbers of the card or cards isolated.[4]

(*e*) From the reference serial number tentatively identify the compound or compounds in the empirical formula-name deck. Locate the corresponding spectrogram in the literature or catalog of spectrograms; otherwise, record the spectrogram of a bona fide sample for matching.

Procedure for Visible and Ultraviolet Technique

7. (*a*) Record spectrograms of the material in original solution and at several successive ten-fold dilutions until the peak of the strongest absorption band lies below an absorbance level of 2.0.

(*b*) Note the spectral position of the prominent absorption bands on all the spectrograms with a precision of 2 mμ. Recall any other characteristics of the material, such as elemental composition or an unusual functional group, that are known without doubt and covered by the codes in the card index.

(*c*) Allowing for error of 2 and 5 mμ in the location of ultraviolet and visible bands, or a spread of 6 and 15 mμ, respectively, sort by machine the appropriate deck of index cards according to directions in the book of codes and instructions.[4]

(*d*) Proceed further as directed in Section 6 (*d*) and (*e*).

APPENDIX

SUPPLEMENTARY DISCUSSION

Machine sorting of the card index saves much time in matching spectral data with spectrograms for qualitative chemical identification and chemical structure correlation. It has many advantages (**9**) over card systems that require hand sorting. The advantage over searching individual spectrograms is tremendous. In an infrared spectroscopic laboratory dealing with a wide variety of substances, the cost of time saved by one or two searches per month can offset the cost of the infrared portion of the card index and machine rental in a year.

Since cards may become lost, damaged, or destroyed, it is wise to purchase or reproduce duplicate cards (Note 2). The original cards may be damaged or destroyed as a result of adverse storage conditions, faulty handling of cards or operation of machinery, or ordinary wear and tear. Duplicate decks of formula - name cards may be stored in two ways for ready hand reference: as received in order of empirical formula or in order of serial number.

NOTE 2.—The cards and codes are copyrighted by the American Society for Testing Materials to protect the system from deterioration through irresponsible reproduction and distribution which would tend to introduce new errors, perpetuate old ones, and prevent proper revisions and additions from being made. However, purchasers of the Wyandotte-ASTM index cards are granted the right to replace lost or damaged cards by reproducing them for their own use only so far as to maintain their decks in working condition. IBM offices offer service or equipment to replace damaged cards.

One of the primary functions of the empirical formula - name index cards is to implement the "guess and check" method of spectrogram identification. Since it requires only about 30 sec to locate the reference to the spectrum of a compound, it is possible to check the spectra of a sizeable number of compounds in a short time. This is a recommended first approach where known information about the unknown material being analyzed would lead one to suspect certain compounds as possibilities. Where little is known about the material being analyzed, a variation of the "guess and check" method may be employed. This involves establishing postulated structures for the unknown via application of structure-frequency correlations as treated by Bellamy (**4**), Jones and Sandorfy (**10**), Colthup (**11**), and others and obtaining the corresponding spectral data through use of the empirical formula - name cards for comparisons with the unknown spectrum. In fact, when any of several conventional approaches (**2**) reaches the point where the spectrum of a given compound is desired for comparison with the unknown, these cards are useful in locating the standard data.

With the spectral data index decks, there are many variations of sorting operations that help to reduce sorting time. For the most part these become apparent to users of the cards and have not been included in the instruction manual of operations. Some of these will be discussed briefly here. The "overpunch" or so-called "y" punch, which indicates very strong bands, lends itself to useful sorts in special cases. Thus, in the spectrum of an unknown mixture, a particularly strong band may obscure a weaker band of another component. A sort, first on the regular code for the strong band, and then a sort of the residue cards on the "y" code for the same region would effect the desired separation. In trial and error sorting for the analysis of mixtures, the special "y" punch can speed up the operation. After a first sort the second sort of the next band can be made more selective by using the "y" code and keeping the cards sorted out if the second band was a strong one, or keeping the rejected cards if the band was a moderately strong one.

Occasionally the spectrum of an unknown displays a lack of bands in regions where most compounds have bands. In such cases, initial sorts to eliminate all cards that do have bands in these positions would be effective. This would automatically be done in the case of mixtures but is not so obvious in the analysis of single component samples.

Users of these cards should be reminded of the flexibility and degrees of reliability built into the Kuentzel system. This begins with initial decisions as to the exact location of a given band. If it is sharp and well-defined, sorting may proceed on a single band position or a very narrow spectrum range. If the band is less well defined and broad, a wider spectrum range can be sorted. One should handle the cards in such a way that if a given operation is not fruitful, one can backtrack to the next previous package of cards for an altered approach without having to return to the bulk of cards in the main decks. This is particularly true if trial sorts based on postulated structures are undertaken. Sorts should be started on the most reliable information and progress toward the less reliable and, where alternate moves of equal merit present themselves, the basic deck of cards from which these experimental sorting sallies are made should be retained as a safeguard against possible failure. The sorting and correlating possibilities coded into these cards are enormous and confidence and dexterity result from continued use.

An apparent limitation of the card index system arises from the large size of the decks and the relatively slow speed of the least expensive card-sorting machines. On the other hand, all of the data are in IBM cards, and the advantages of manipulation by more complicated IBM equipment and services for data storage and rapid retrieval are quite attainable, if frequency of use and economics justify. With the least expensive sorters, the speed of handling and sorting averages about 500 cards per minute, whereas the infrared and other spectral data cards number more than 37,000 and 14,000 respectively, in each of the spectral data and formula-name decks. To reduce the number of cards to be handled in a search with an inexpensive sorter, it may be desirable to "presort" the cards into smaller decks according to some selected criterion, such as strongest absorption band, elemental analysis, etc. The cards have extra space for coding by punch marks any desired arrangement of the index.

By filing the cards in presort arrangements based on characteristics of spectra, an initial sort results from the act of removing the cards from the file. One such system makes use of the three strongest bands and assigns each card to a group that signifies the presence of a strong band in a given region of the spectrum. The same card is placed in each of the three groups determined by the three selected for sorting. Cards are then filed by groups and an initial sort results from the act of selecting a group with which to begin the search.

The chemical classification code adds great utility to these cards. One obvious use is the establishment of structure - band correlations. It is a simple operation to obtain cards indexing all compounds that have an absorption band at any given position of the spectrum and then determine the percentage of these which index compounds having a given structure, or to apply the reverse procedure of segregating cards indexing compounds of like structure and searching for bands in common. Details of the codes and search procedures for structure studies are given in the book of codes and instructions.[4]

Combination sorting on both bands and structure is recommended whenever reliable partial knowledge of the nature of the unknown warrants. Where this is a common occurrence, it is suggested that permanent separate files of cards based on types of structure or classes of compounds be maintained. Much of this can be done by machine.

A suggested grouping system is based on the breakdown of structural types used by Bellamy (**4**) which separates the cards on the basis of elements involved such as (*1*) carbon and hydrogen only; (*2*) carbon, hydrogen, and oxygen only; (*3*) carbon, hydrogen, and nitrogen only; etc. An elemental analysis dictates which deck to use in this case. Another system divides the cards on the basis of whether there is, or is not, a strong band in the 5 to 6-μ region. One then sorts only the appropriate deck to match an unknown spectrum. Others file separate decks indexing the spectra of inorganics, solids, gases, and liquids or solutions. Elaborate systems of presort decks based on classes of compounds or the presence or absence of bands in selected regions of the spectrum have been reported by users of the cards. Finally, a laboratory that does considerable repetitious analytical work merely places all cards indexing materials that have been analyzed into a separate deck. In all cases, use of such presort decks greatly reduces sorting time.

Presorted decks are available for a fee by special arrangement through ASTM Headquarters. The presorted decks may derive from the complete decks of spectral data or formula - name cards, or any selected portion. Such special decks are made to the customer's specifications to index (*1*) spectra of particular classes of compounds, (*2*) spectra of compounds having particular structural features or combinations of elements, (*3*) spectra showing particular band groupings, etc.

The card index will grow and become more comprehensive only if laboratories continue to exercise diligence in submitting their spectrograms for publication. Publication in a widely distributed journal or in the catalogs of one of the organizations listed in Table II is appropriate. Some of the latter organizations will record spectrograms of pure compounds on loan. In any case, contact should be made in advance with the publisher for instruction on policy, formats, etc.

There is also continued need for volunteers to code published spectral data for the card index. Laboratory organizations are urged to sponsor their personnel in this activity. Participation in this activity leads to greater familiarity with spectral data and, at the same time, serves the field of spectroscopic science. Volunteers for this service should communicate with ASTM Headquarters.

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REPORT OF COMMITTEE E-15
ON
ANALYSIS AND TESTING OF INDUSTRIAL CHEMICALS*

Committee E-15 on Analysis and Testing of Industrial Chemicals held two meetings during the year: on June 30 and July 1, 1960, in Atlantic City, N. J., and on Feb. 2–3, 1961, in Cincinnati, Ohio. A special meeting of the officers was held on January 13, 1961, in Philadelphia, Pa.

The committee consists of 142 individuals representing 89 companies and organizations, 14 other committees of the Society, and 6 consulting members. Due to the widespread interest in chemicals, the objectives of the committee have been outlined in a letter to the officers of all technical committees of the Society, inviting liaison with them in areas of common interest. The committee's organization was shown in its first annual report issued last year.

Gas chromatography had been suggested as an area for work in the committee, but a special meeting held jointly with representatives of other interested groups on July 1, 1960, led to the organization on February 6, 1961, of a separate new Committee E-19 on Gas Chromatography with about 125 participating individuals.

Analysis of radioactive chemicals also had been suggested for work in the committee, but consideration of this subject led to the recommendation that it be handled in Committee E-10 on Radioisotopes and Radiation Effects with such assistance as might be needed from Committee E-15.

Active participation in ISO/TC 47 on Chemicals (particularly methods of analysis for industrial chemicals) was voted by the Committee. The Board of Directors of the Society has approved the organization of a National Committee under the sponsorship of Committee E-15 for work with the American Standards Assn.

Work in the new areas of chelating agents and *n*-butyllithium was authorized by the Executive Subcommittee and organization of work groups is in progress.

Some 30 chemical analytical methods prepared for their products by other ASTM committees have been referred to Committee E-15 for review.

NEW TENTATIVE

The committee recommends for publication as tentative the proposed Recommended Practice for Developing Precision Data on ASTM Methods for the Analysis and Testing of Industrial Chemicals as appended hereto.[1]

This recommendation has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee 2 on Standards and Reagents (S. M. Tuthill, chairman) has drafted methods for preparing and standardizing NaOH, HCl, and H_2SO_4 solutions, with particular attention to the needs of Subcommittees B-1 on Mineral Acids and B-2 on Alkalies, and is circulating these methods for comment. Methods for preparing and standardizing $AgNO_3$ and ammonium thiocyanate have been requested, and a draft of these has been prepared. This subcommittee expects to serve as the clearing house for reagents and standards needed by the other subcommittees of Committee E-15. Liaison has been established with the task group on uniformity of reagents for Committee D-19 on Industrial Water and this will facilitate efforts to adopt uniform practices within ASTM.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] The new tentative appears in the 1961 Book of ASTM Standards, Part 8.

[2] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee 3 on Precision and Accuracy (R. J. Sobatzki, chairman) published for information with last year's annual report, a glossary for a Proposed Recommended Practice for Developing Precision Data on ASTM Methods for the Analysis and Testing of Industrial Chemicals. The subcommittee now has completed the other sections of this recommended practice including: *Section B.*—Planning the Inter-Laboratory Study, *Section C.*—Statistical Design of Collaborative Data, *Section D.*—Format of Precision Statements, *Section E.*—Bias (Systematic Error), and *Section F.*—Presentation of Data. The committee is recommending the recommended practice for adoption as tentative. The subcommittee has cooperated with Committee E-11 on Quality Control of Materials.

Subcommittee 4 on Sampling (H. O. Hehner, chairman) has been aided by an extensive search of the literature on sampling, from 1937 to the present, sponsored by the Monsanto Chemical Co. at Battelle Memorial Institute. The sources include general analytical books, technical journals, practices of technical organizations, manufacturers of sampling devices, and methods of companies of this subcommittee's members. Complete articles have been microfilmed and xeroxed, and now are being classified and evaluated for applicability to chemical products. Because of the magnitude of the undertaking, initial effort is being concentrated in two task groups on (1) bulk sampling of simple single-phase liquids, and (2) bulk sampling of free-flowing finely divided solids.

Subcommittee A-1 on Elements (Leavitt Gard, chairman) has organized two task groups:

(1) The Task Group on Chlorine and Sulfur (A. P. Scanzillo, leader) has completed a comprehensive survey of methods for determining chlorine and sulfur by Parr bomb fusion. First and second drafts of proposed methods have been circulated and balloted and the task group is preparing third drafts for collaborative study.

(2) The Task Group on Phosphorus and Silica (D. K. Bannerjee, leader) has prepared a bibliography on methods for phosphorus and silicon by Parr bomb fusion, and drafts of methods for these elements are being studied with plans for collaborative testing.

Subcommittee A-2 on Functional Groups (J. Mitchell, Jr., chairman) has organized three task groups:

(1) The Task Group on Determination of Hydroxyl Groups in Aliphatic Organic Compounds (T. P. Callan, leader) has circulated drafts of a general acetic anhydride method and a perchloric acid catalyzed modification of this method for collaborative studies, using pentaerythritol, ethylene glycol, dodecanol, and nonyl phenol as representative hydroxyl-containing compounds. Copies also were sent to following subcommittees of Committee D-1: Subcommittee V, Group 3 on Pentaerythritol, and Subcommittee II which is working on a hydroxyl method for drying oils. Comments

were exchanged with these subcommittees. The task group expects to have a final draft of the acetic anhydride method ready for consideration by the committee in June. More information is needed on the acid-catalyzed modification before a final draft can be prepared.

(2) The Task Group on Unsaturation (E. N. Luce, leader) has been studying the pyridine sulfate dibromide method with particular consideration being given to the model compounds methyl methacrylate, mesityl oxide, cyclohexene, and undecenoic acid, which have been shown to react essentially quantitatively. A final draft of this method is expected to be ready for consideration by the committee in June.

(3) The Task Group on Organic Peroxides (D. K. Bannerjee, leader) held its first meeting in Cincinnati, Ohio, and selected as its objectives: (*a*) analytical methods for assay, (*b*) analytical methods for determining low concentrations of peroxides, (*c*) information on the formation of peroxides, particularly in solvents, and (*d*) hazards and safe practices in handling peroxides. Work has been started on a bibliography of the analytical methods of interest to the task group and it is expected to have one assay method and one low level concentration method available for comment in June.

Subcommittee A-3 on Physical Properties (W. A. MacNaughton, chairman) has two active task groups:

(1) The Task Group on Density-Volume Relationships for Industrial Liquid Chemicals (B. H. Cummings, leader) has prepared a Proposed Method for Calculation of Volume and Weight of Industrial Chemical Liquids, including tables for the following ten chemical products: isopropyl ether, hexylene glycol, ethyl alcohol, epichlorohydrin, mesityl oxide, 95 per cent by weight glycerin, 96 per cent by weight glycerin, allyl chloride, allyl alcohol, and diacetone alcohol. The method and tables are being distributed to the members of the subcommittee for study and acceptance prior to submittal to the committee. Similar tables are needed for many such products and the task group plans to extend the tables to cover additional commercial liquid chemicals handled in large quantities. Overlap of work of other committees such as the Method for Calculation of Volume and Weight of Benzene, Toluene, and Paraxylene (D 1555–58 T) prepared by Committees D-2 on Petroleum Products and D-16 on Industrial Aromatic Hydrocarbons, will be avoided through proper liaison.

(2) The Task Group on Melting Points and Freezing Points (W. A. MacNaughton, leader) has written procedures for capillary and hot stage melting point determinations, and these are being evaluated in a number of laboratories. The Method of Test for Solidification Point of Aromatic Hydrocarbons and Related Materials (D 1493–58 T) appears to be generally applicable to chemical products and is being studied by the task group. If favorably considered, it is planned to request Committee D-16 to amend the title and scope of this method to indicate its broader application.

Need for study and establishment of methods for color of industrial chemicals has been discussed but organization of a task group on color has been deferred pending selection of a leader.

Subcommittee A-4 on Water (W. W. Becker, temporary chairman) has reviewed the several ASTM methods for determination of water using the Karl Fischer reagent. Task groups under the leadership of C. M. Gambrill for reagents, J. Mitchell, Jr. for interfering groups, and E. G. Wiest for apparatus, have prepared comprehensive descriptions of these sections, and the subcommittee is assembling them into a recommended practice or general method applicable to chemical products. Much helpful information for other ASTM committees will be contained therein.

Subcommittee B-1 on Mineral Acids (W. Clavan, chairman) has organized two task groups:

(1) The Task Group on Sulfuric Acids (N. C. Schultze, leader) is considering methods for total acidity, specific gravity, nonvolatiles, iron, and nitrates in sulfuric acid. From collaborative tests using four different procedures, a total acidity method has been selected and is being prepared for final evaluation. Drafts of methods for the other determinations have been prepared and discussed, and revisions based on comments of the subcommittee are being incorporated for collaborative evaluation.

(2) The Task Group on Hydrochloric Acid (R. E. Richard, leader) has prepared drafts of methods for total acidity, Baumé gravity, ash, sulfur, free chlorine, and heavy metals. These have been discussed and will be rewritten for consideration by the subcommittee. Methods for iron and color will be added.

Subcommittee B-2 on Alkalies (R. J. Funston, chairman) has prepared methods for determination of total alkalinity, carbonate, chloride, sulfate and iron in sodium hydroxide, and has carried out preliminary collaborative tests. Necessary changes are being made in the methods on the basis of the results obtained and plans are under way for establishment of their precision and accuracy on various strengths of sodium hydroxide. Because of its importance for meaningful results, instructions for sampling sodium hydroxide also are being prepared for inclusion with the proposed methods and review by Subcommittee 4.

Methods for other alkalies are on the agenda for future consideration.

Subcommittee B-3 on Alcohols (W. R. Deitz, chairman) has organized two task groups:

(1) The Task Group on Ethylene Glycol (R. L. Anderson, leader) has carried out cooperative tests on methods for determination of specific gravity, distillation range, acidity, and color and is studying the results. A first draft of a method for iron determination is under revision and a potentiometric method for determining chloride is being investigated.

(2) The Task Group on Methanol (J. L. Carlson, leader) is reviewing the methods of sixteen different companies for selection of the tests desired for standardization. Present ASTM methods will be used wherever applicable.

This report has been submitted to letter ballot of the committee, which consists of 142 individuals; 95 returned their ballots, of whom 93 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

W. A. KIRKLIN,
Chairman.

R. C. JOHNSON,
Secretary.

REPORT OF COMMITTEE E-17
ON
SKID RESISTANCE*

Committee E-17 on Skid Resistance held two meetings during the year: in Atlantic City, N.J., on June 28, 1960, and in Cincinnati, Ohio, on Feb. 2, 1961. At the June meeting, officers were elected and a scope for the committee was adopted. The February meeting was the first working meeting of the committee. At this time appointments were made to a number of subcommittees, scopes were assigned to these subcommittees, and each subcommittee held a meeting.

The committee lost one member by death: Earl J. Felt. In recognition of the valuable contributions of Mr. Felt in the field of research on skid resistance and of the loss which members felt in his passing, an appropriate memorial resolution was prepared and adopted and is being entered on the permanent records of the committee and the Society.

Although the committee has been active for less than one year, some accomplishments have already been made. A series of cooperative tests are being performed by various agencies on a variety of specimens developed under the sponsorship of Subcommittee II-d on Standard Test Surfaces in an effort to develop a specimen or specimens which may be used repeatedly for calibration of slipperiness testing equipment and to permit comparison of different devices of this nature. Subcommittee II-e on Tire Characteristics and Significance has produced a tentative specification for a standard slipperiness test tire, and at least one tire manufacturer has agreed to produce this tire.

Respectfully submitted on behalf of the committee,

E. A. WHITEHURST,
Chairman.

J. H. DILLARD,
Secretary.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

REPORT OF COMMITTEE E-19
ON
GAS CHROMATOGRAPHY*

The organizational meeting of Committee E-19 on Gas Chromatography was held on Feb. 6, 1961, in Philadelphia, Pa. At that time officers were elected and an Interim Executive Committee was appointed pending the completion of the permanent Executive Committee and the adoption of by-laws by the members. The purpose of the committee is the promotion of the development of gas chromatography by : (1) coordinating scientific applications and methods of analysis based on gas chromatography in cooperation with other technical committees of the Society and other organizations; (2) cooperating with national and international bodies in standardizing presentation of data and nomenclature; (3) developing general methods and other appropriate standards; (4) providing a forum for exchange of information; (5) stimulating research in this field; and (6) collecting and disseminating critical data.

The permanent Executive Committee has now been completed to consist of the following:

Chairman, B. F. Dudenbostel, Jr., Esso Research and Engineering Co.
First Vice-Chairman, C. H. Orr, Proctor & Gamble Co.
Second Vice-Chairman, N. Brenner, Perkin-Elmer Corp.
Secretary, C. G. Harriz, Houdry Process Corp.
Chairman, Subcommittee I on Definitions, Nomenclature, and Standard Data, B. O. Ayers, Phillips Petroleum Co.
Chairman, Subcommittee III on Research, M. K. Testerman, University of Arkansas.
Chairman, Subcommittee IV on Coordination and Standardization of Methods, W. C. Jones, Jr., Humble Oil & Refining Co.
Members-at-Large, W. W. Brandt, Kansas State University and D. H. Desty, British Petroleum Co.

In addition to serving as Vice-Chairmen, C. H. Orr and N. Brenner will also act as Co-chairmen of Subcommittee II on Programs and Papers.

As an initial activity, Committee E-19 held an informal discussion forum in Chicago, Ill. on June 9, 1961, under the sponsorship of Subcommittee I on Definitions, Nomenclature, and Standard Data. A panel consisting of H. M. McNair, Esso Research and Engineering Co.; D. D. DeFord, Northwestern University; R. A. Dinerstein, American Oil Co.; and D. H. Desty, British Petroleum Co., led discussions on qualitative identifications in chromatography; presentation of retention parameters and additional column data required for column optimization; adsorption of solute on liquid surface; specification of sensitivity, time constant, etc., for chromatographic detectors; and the experience of European groups working on standard data presentation.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Definitions, Nomenclature, and Standard Data (B. O. Ayers, chairman) plans to establish two task groups, one to study definitions and nomenclature and another to study methods of standard data presentation. Comments and suggestions in areas of concern to Subcommittee I will be solicited from its members and interested persons. It is hoped that by the end of 1961, a summary of the response to this solicitation and of a complete literature survey will be completed. At that time preliminary recommendations for subcommittee action will be made.

Subcommittee II on Programs and Papers (C. H. Orr and N. Brenner, cochairmen) is assisting in plans for a meeting of Committee E-19 in the Midwest during September or October, 1962. It is also possible that Committee E-19 may serve as co-sponsor with the Instrument Society of America for the 1963 East Lansing Gas Chromatography Symposium.

Subcommittee III on Research (M. K. Testerman, chairman) held its first meeting on June 9, 1961, at Chicago, Ill. A brief discussion on the scope of the activities was presented by the chairman. It was announced that a poll would be made of interested persons on what activities they might feel important. Also noted was the fact that a close liaison between Subcommittees I and III is necessary due to the high degree of art that still exists in this field of instrumentation. It was established that one of the first activities of Subcommittee III will be that of an informal forum consisting of interim or progress reports (not normally suitable for formal presentation) on new concepts, unique instrumentation, and new techniques in the field of gas chromatography. This forum will be held during each annual meeting of Committee E-19.

Harold M. McNair of Esso Research and Engineering Co. presented a stimulating talk on "Speculations on the Future of Gas Chromatography," followed by floor discussion.

Subcommittee IV on Coordination and Standardization of Methods (W. C. Jones, Jr., chairman) held its organizational meeting on June 9, 1961, in Chicago to discuss the scope and method of operation of the subcommittee, especially with respect to other ASTM groups having mutual interests. After discussion, the establishment of three task groups was approved with the following objectives: (1) to select the correct format for gas chromatography methods, (2) to review methods on gas chromatography, and (3) to write methods concerning general techniques. The chairman and memberships of these task groups will be announced when appointed.

Respectfully submitted on behalf of the committee,

B. F. DUDENBOSTEL, JR.,
Chairman.

C. G. HARRIZ,
Secretary.

REPORT OF COMMITTEE F-1
ON
MATERIALS FOR ELECTRON TUBES AND SEMICONDUCTOR DEVICES*

Committee F-1 on Materials for Electron Tubes and Semiconductor Devices held three meetings during the year: in Skytop, Pa., Nov. 3 and 4, 1960; in Washington, D. C., Feb. 25 and 26, 1961; and June 26 and 27, 1961 in Atlantic City, N. J.

The committee membership totals 96 members, of whom 42 are classified as producers, 36 as consumers, and 18 as general interest members.

M. H. Batlan was appointed chairman of Subcommittee II on Insulators, replacing C. M. Harman, who resigned. A. S. Rugare was appointed secretary of Subcommittee VI on Semiconductors, replacing W. A. Jemain; and N. L. Hobbs was appointed secretary of Subcommittee X on Control of Contaminants. H. Stern was appointed as Committee F-1 liaison member with Committee E-10 on Radioisotopes and Radiation Effects.

Stanton Umbreit was presented the ASTM Award of Merit at the 1960 Annual Meeting.

Highlighting the activities of the past year was the committee's consideration of the use of the metric system in its standards. It is intended that both English and metric systems will be used in current Committee F-1 standards. While this is a long-term project, it takes cognizance of a worthwhile trend. The committee recognizes that the effort must be well known and that adequate time, probably several years, must be provided for its acceptance.

Of particular significance was the Symposium on Materials and Electron Device Processing conducted under the auspices of Subcommittee X and held in Philadelphia on April 5, 6, and 7, 1961. The sessions were attended by over 300 interested technical people and provided a program of 35 papers on this important subject. The papers have been published as *ASTM STP No. 300*.

With respect to standardization work of Subcommittee VI on Semiconductors, a strengthened liaison with the National Bureau of Standards via Messrs. Wildhack, French, and Marsden was established.

Recommendations Accepted by the Administrative Committee on Standards

Subsequent to the 1960 Annual Meeting, Committee F-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specifications for:

Mica Bridges for Electron Tubes (F 12 - 60 T), and

Disk Cathode Assemblies (F 13 - 60 T).

These recommendations were accepted by the Standards Committee on Sep-

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***** Sixty-fourth Annual Meeting of the Society June 25-30, 1961.

tember 29, 1960, and the new tentative specifications appear in the 1960 Supplement to Book of ASTM Standards, Part 2.

NEW TENTATIVES

The committee recommends for publication as tentative the following recommended practice, specification, and method as appended hereto:[1]

Tentative Recommended Practice for:

Making and Testing a Reference Glass-Metal Bead-Seal.

Tentative Specification for:

Iron-Nickel-Cobalt Sealing Alloy.

Tentative Methods of:

Mechanical Test for Measuring Diameter or Thickness of Wire and Ribbon for Electronic Devices and Lamps.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Specification for Aluminum Oxide Powder (F 7 – 58 T):[2]

Section 2.—Revise the table of chemical requirements to read as follows:

Alumina (Al_2O_3), min, per cent	99.65
Silica (SiO_2), max, per cent	0.08
Magnesia (MgO), max, per cent	0.08
Ferric oxide (Fe_2O_3), max, per cent	0.10
Total alkali (as Na_2O), max, per cent	0.06
Carbon (C), max, per cent	0.01
Calcium oxide (CaO) max, per cent	0.08
Zirconium oxide (ZrO_2) max, per cent	0.08
No other single impurity to exceed, per cent	0.03
Water-Soluble (as NaCl), max, ppm	300

Section 4.—Revise the heading to read "Sampling" and delete the second sentence.

Section 5(b).—Change to read as follows:

> (*b*) *Water-Soluble Content*—The total water-soluble content shall be determined by measuring the electrical conductivity of a 100-ml distilled or deionized water extract from a 5 g sample (or other convenient size). Elutriation shall consist of vigorously shaking the alumina suspension in a flask for 1 to 2 min, allowing the solids to settle, and decanting the clear supernatant liquid after 30 to 60 min, the conductivity of which is measured with a standard cell and bridge. This measurement shall be corrected for the "blank" reading on a similarly treated equal volume of the same pure water. The net increase in conductivity of the water extract shall be first converted to parts of water-soluble content as NaCl per million of water (factor is 1 micromho = 0.5 ppm NaCl), then related to the actual weight of elutriated alumina. For example, on a typical 100-ml extract 10 micromhos (net) or 5 parts of NaCl per million of water in this volume of liquid is equivalent to 100 parts of NaCl per million of alumina on the basis of a 5-g sample.

Tentative Specification for Nickel Alloy Cathode Sleeves for Electronic Devices (F 239 – 57 T):[2]

Section 1.—Change the scope to read as follows:

> 1. This specification covers nickel alloy cathode sleeves, seamless, welded-and-drawn, lockseam, or lapseam, for use as indirectly heated oxide-coated cathodes in electron devices.

Section 6.—Add a new Paragraph (*h*) to read "(*h*) *Seam.*—The seam dimensions shall be in accordance with the values listed in Table VII."

Table I.—Make the following revisions in Table I for alloy grades 3, 10, and 11, and add a new Footnote *c*:

Grade	Copper, max, per cent	Iron, max, per cent	Magnesium, per cent	Titanium, per cent	Carbon, per cent[c]
3 ...	0.10	0.10	0.01 to 0.08	0.01 to 0.05	...
10 ...	0.10	0.10	0.01 to 0.10	0.005 max	...
11 ...	0.10	0.10	0.01 to 0.08	0.01 to 0.05	...

[c] Carbon limits specified apply to cathode material only. Carbon content is higher for large tubing or strip for rerolling or redrawing.

Table II.—Add to the portion of Table II on Circular Cross-Section

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[1] The new tentatives appear in the 1961 Book of ASTM Standards, Part 2.

[2] 1958 Book of ASTM Standards, Part 2.

Sleeves the following: "Lapseam: 0.025 to 0.040, incl....±0.0005."

Table III.—Under Circular Cross-Section Sleeves, add "Lapseam: Up to 0.040, incl.... $D - (3W + 0.0014)$."

Under Shaped Cross-Section Sleeves, add "Lapseam: All sizes.......... $M - (2W + 0.0016)$."

To Footnote *a*, add "M = specified minor outside diameter in inches."

TABLE I.—PERMISSIBLE VARIATIONS IN LENGTH.

(Revision of Table V, Specifications F 239)

Specific Length, In.	Permissible Variations in Specified Length, in.		
	Seamless, or Welded-and-Drawn	Lockseam	Lapseam
Under 1......	±0.005	±0.005	±0.005
1 to 2........	±0.005	±0.010	...
Over 2.......	±0.010	...	...

Table IV.—Add a new column for Lapseam to read "0.0001" for wall thickness of 0.0015 to 0.0029, excl.

Table V.—Change to read as shown in the accompanying Table I.

Table VI.—Add to Table VI, "Lapseam: All sizes" with the following tolerances:

Tab Location.........	180 ± 15 deg to seam
Width, Flattened, in....	0.010 to 0.015
Permissible Variation, in..................	±0.003
Tab Length...........	1 to 15 ± 0.5 mm
Tab Radius, in........	0.010 to 0.015

Add tab radius of "0.010 to 0.15" to all categories of Table VI.

New Table VII.—Add a new Table VII as shown in the accompanying Table II.

Tentative Method of Testing Sleeves and Tubing for Radio Tube Cathodes (F 128 - 56 T)[2]: Revise as appended hereto.[3]

Tentative Recommended Practice for Cathode Melt Prove-In Testing (F 238 - 55 T)[2]: Revise as appended hereto.[3]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

TABLE II.—PERMISSIBLE VARIATIONS IN SEAM WIDTH AND THICKNESS

(New Table VII, Specifications F 239)

LOCKSEAM

Nominal Wall Thickness, in.	Permissible Variations, in.	
	Seam Width	Seam Thickness, max
0.0021.........	0.020 to 0.025	0.0098
0.0025.........	0.020 to 0.025	0.0125
0.003..........	...	0.0153
0.0035.........	...	0.0190

LAPSEAM

Lap opening 0.0005 max.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Cathode Materials (A. P. Haase, chairman).—The subcommittee comprises three principal task groups whose work over the past year has highlighted investigations of methods of measuring cathode temperature in the vacuum tube with an improved degree of accuracy, culminating in a recommended practice for the measurement of temperature of thermionic emitters, currently ready for committee approval.

The Interface Task Group modified the scope of Method F 300 for committee approval. Improvements to methods of calibration and evaluation of interface impedance are the main points. The preparation of a proposal for an International Electrotechnical Commission recommendation on interface impedance measurement was assigned to the U. S. Such a document has been prepared in IEC format by the ASTM in cooperation with the U. S. National Committee.

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[3] The revised tentatives appear in the 1961 Book of ASTM Standards, Part 2.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Important revisions of Method F 128, particularly in the methods of collapse strength testing, were made by the physical and chemical test section. The results of these efforts will provide desired limits for Specification F 239.

Factory melt prove-in tests were made, the first report of the period being on Cathaloy A31 material.

New areas of testing coating adherence using standard scratch test were under investigation as applicable to cathode coatings. Interlaboratory tests are in progress.

Subcommittee II on Insulators (M. H. Batlan, chairman) is conducting an investigation of milled alundum. Early reported findings show the effect of low-pH and high-pH material. Further reports indicated that the pH and coating effects were tied to steel-ball grinding media versus ceramic-ball milled media. This investigation will be further strengthened by electron microscope studies now being made, together with an investigation of spectrographic differences of the alundums.

The subcommittee anticipates further revisions in Methods F 652, particularly in the area of use of an automatic device for lowering the pin of the tapered pin gage and the use of special overscreen charts or reticles.

The group expects to add recommended practices on mica design for Specification F 12 on Mica Bridges for Electron Tubes. Consideration is being given to writing a specification on the use of integrated and reconstituted mica as a substitute for natural mica bridges. This subcommittee finished its work on the revision of Specification F 7 as noted earlier in this report.

Subcommittee III on Strip (P. P. Prichett, chairman) has been engaged in the measurement of gas content of typical anode materials with the aim of developing a test method.

In addition, the investigations of surface quality measurements resulted in interlaboratory tests being set up, principally for reflectometer tests. Other means of measuring matte and bright surface finishes are being investigated.

This subcommittee is coordinating with Subcommittee V in regard to tolerance requirements of strip in connection with the iron-nickel-cobalt sealing alloys. Still in the discussion stages are projects involving high-conductivity anode strip, close-tolerance strip measurement, and changes in the chemical composition of nickel strip to establish better agreement between Specifications F 1, F 2, F 3, F 4, and F 239.

Subcommittee IV on Wire (D. R. Kerstetter, chairman) has been revising Specifications F 288 and F 290 for committee balloting at the fall meeting.

Other work being done by this subcommittee includes tensile evaluation of lead weld knots, the review of Specification F 9 on side rod wire to cover materials currently in use, and the review of Method F 113 on bend testing of support wires to include use of standards for calibrating the bend tester.

It is intended to solicit the interest of various semiconductor manufacturers in covering the various wire and ribbon materials now in use on semiconductors in this committee.

Subcommittee V on Metallic-Nonmetallic Seals (V. J. DeSantis, chairman) has completed a specification on iron-nickel-cobalt alloys for sealing to glass and has conducted investigations on a proposed method for tensile and vacuum testing of metallized seals. Other activities include interlaboratory tests on iron-nickel alloys, principally aimed at evaluation of expansion and chemical composition as appropriate to a sealing alloy. Reference seals for the metal-glass application are under consideration, and also the preparation of Dumet color standards.

Subcommittee VI on Semiconductors (H. M. Pollack, chairman) has completed a method of test for the determination of electrical resistivity for semiconductor materials. Studies are under way of dislocations in silicon, germanium dioxide, crystallographic orientation, and oxygen in silicon, the results of which will be applicable to future specification or method-of-test work.

Subcommittee VIII, Editorial (E. A. Thurber, chairman) has provided for uniformity of terms, format, and beneficial recommendations as applied to all the specifications generated within the committee. The chairman in particular participated in depth in appraising the applicability of the metric system as a long-term benefit to specifications and instituted the current program of having all specifications appear in both systems as they originate in the committee, with the English system taking precedence whenever a dispute on conversion factors occurs. The committee appreciates that however beneficial the conversion to the metric system may be, it is a program of several years duration and must be done without embarrassment to existing supplier-consumer relationships. This effort is coordinated to the JEDEC groups through Committee F-1 representative W. Hindman.

Subcommittee IX on Materials Analysis (J. C. Hickle, chairman) has participated in liaison activities with the other subcommittees in their needs for analytical procedures. Principal efforts were in the fields of improved chemical and spectrographic analyses as applied to the problems of Committee F-1 and working through appropriate liaison with the E Committee groups.

The expressed difficulties in determining small quantities of magnesium in nickel occasioned investigation of a photometric method of Andrew and Gentry (Mullard) of England, which appears to be a desirable alternate for the Luke oxine procedure. In investigations for methods of copper in nickel, interlaboratory tests using the photometric neocuproine method were satisfactory to occasion the subcommittee's recommendation for this method to appear in the report of Committee E-3 on Chemical Analysis of Metals for incorporation into the new ASTM Methods of Chemical Analysis of Metals. The program of determining cobalt in nickel has not concluded but is concerned with photometric and polarographic methods.

Significant discussions have also taken place with respect to tests for rare earths in nickel and the application of X-ray fluorescence analysis to tungsten-nickel alloys.

Subcommittee X on Control of Contaminants (D. E. Koontz, chairman) has had a most active year, with a particularly high growth rate. The meetings during the period were largely concerned with background discussions to provide a satisfactory basis for future methods of test and specifications. Currently the work of the committee includes the categories of airborne contaminants, surface contaminants, and the examination of solvents.

The background work on airborne contaminants includes microscopic examination, settling and filtration, and impact methods, as well as the photoelectric method approach. The surface contaminants effort has concerned itself with conductivity measurements, wettability tests, microscopy of surface particle identification, radioactive tracer tests, and surface potential tests. The examination of solvents work includes investigations on conductivity, biological impurities, evaluation of total solvents, method of test for organics, etc.

Highlighting this subcommittee's work was the Symposium on Materials and Electron Device Processing mentioned earlier in this report.

This report has been submitted to letter ballot of the committee, which consists of 96 members; 63 members returned their ballots, of whom 42 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

S. A. STANDING,
Chairman.

C. L. GUETTEL,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee F-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method for:

Evaluation of Glass-to-Metal Headers for Electron Devices (F 18 – 61 T), and
Tension and Vacuum Testing Metallized Ceramic Seals (F 19 – 61 T).

Revision of Tentative Method of Test for:

Sublimation Characteristics of Metallic Materials by Electrical Resistance (F 278 – 55 T), and
Interface Impedance Characteristics of Vacuum Tube Cathodes (F 300 – 57 T).

Revision of Tentative Specifications for:

Tungsten Wire for Electronic Devices and Lamps (F 288 – 57 T).

Revision and Reversion to Tentative of Standard Methods for:

Measuring Mica Stampings or Substitutes Used in Electronic Devices and Lamps (F 652 – 51).

These recommendations were accepted by the Standards Committee on September 12, 1961, and the new and revised tentatives appear in the 1961 Book of ASTM Standards, Part 2.

On January 3, 1962, the Administrative Committee on Standards accepted the following recommendations submitted by Committee F-1 which are available as separate reprints:

New Tentative Specification for:

High Conductivity Composite Aluminum-Steel-Copper for Electron Tubes (F 20 – 62 T).

New Tentative Method of Test for:

Hydrophobic Surface Films (the Atomizer Test) (F 21 – 62 T), and
Hydrophobic Surface Films (Water Break Test) (F 22 – 62 T).

New Tentative Method for:

Measuring and Counting Particulate Contamination on Surfaces (F 24 – 62 T).

New Tentative Recommended Practice for:

Temperature Measurement of Thermionic Emitters (F 23 – 62 T).

Revision of Tentative Specification for:

Nickel Clad and Nickel-Plated Steel Strip for Electron Tubes (F 1 – 57 T),
Aluminum-Clad Steel Strip and Nickel-Steel-Aluminum Composite Strip for Electron Tubes (F 2 – 57 T),
Nickel Strip for Electron Tubes (F 3 – 57 T),
Carbonized Nickel Strip and Carbonized Nickel-Plated and Nickel-Clad Steel Strip for Electron Tubes (F 4 – 57 T),
Nickel Alloy Cathode Sleeves for Electron Devices (F 239 – 61 T), and
Round Wire for Use as Grid Lateral Winding Wire in Electron Tube Grids (F 290 – 57 T).

REPORT OF COMMITTEE F-2
ON
FLEXIBLE BARRIER MATERIALS*

Since its last report to the Society in June, 1958, Committee F-2 on Flexible Barrier Materials has held six meetings: on October 15, 1958, and April 15, 1959, in Chicago, Ill.; Nov. 19, 1959, in New York, N. Y.; April 7, 1960, in Atlantic City, N. J.; Nov. 3, 1960, in New York, N. Y.; and on March 8, 1961, in East Lansing, Mich.

The committee consists of 80 voting members, of whom 46 are classified as producers, 16 as consumers, and 18 as general interest members.

NEW TENTATIVE

The committee recommends for publication as tentative the Definitions of Terms Relating to Flexible Barrier Materials as appended hereto.[1]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I, Executive Committee (C. C. Sutton, chairman) held a meeting at the Division of Food, USDA, on Sept. 16, 1960, in Washington, D. C., to establish liaison toward the development of extraction methods for flexible barrier materials acceptable to the Food & Drug Administration. A brochure to publicize the activities of the committee was printed and made available to all interested parties. The desirability of sponsoring research on gas transmission methods for flexible barrier materials has been taken under consideration.

Subcommittee II on Nomenclature and Definitions (H. H. Haines, chairman) has defined 4 terms mentioned earlier in this report.

Subcommittee III on Testing Methods (K. W. Ninnemann, chairman) established a new section on Extraction Test Methods and is exploring the possibility of developing new test methods for cellulosic films and various laminates.

Section on Extraction Test Methods (I. F. Peake, chairman) has reviewed all extraction methods applying to flexible barriers and is concentrating its efforts on three procedures. Interlaboratory studies of these methods have been initiated.

Section on Physical Test Methods (G. R. Rugger, chairman) is subjecting static and dynamic loading procedures for determining seal strength to interlaboratory testing. Impact tests applicable to the broad area of flexible barriers are being evaluated. The properties necessary to evaluate stiffness and available methods and apparatus are being studied.

Section on Transmission Test Methods (W. B. Kunz, chairman) is developing water vapor and gas transmission methods for regenerated cellulosic materials. Existing methods and new approaches are being investigated for flexible barrier composites.

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* Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] The new tentative appears in the 1961 Book of ASTM Standards, Part 6.

Subcommittee IV on Specifications (Paul Cope, chairman) has listed and defined all of the properties of flexible barrier materials that must be characterized for a specification.

The subcommittee has explored the possibility of developing toughness as a specifiable property of flexible barriers. A survey has indicated that its units may be expressed as foot-pounds per square foot, the work necessary to break the material. An interlaboratory study of the toughness of four types of flexible barriers has begun.

A new section was authorized to explore regenerated cellulosic films.

Subcommittee V on Liaison (J. W. Goff, chairman), has developed active liaison with appropriate ASTM Committees. Pertinent work of these committees as well as that of the Packaging Institute and the Technical Assn. of the Pulp and Paper Industry is brought to the attention of the committee.

This report has been submitted to letter ballot of the committee, which consists of 80 members; 42 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

C. C. SUTTON,
Chairman.

R. H. GILJOHANN,
Secretary.

TECHNICAL PAPERS

With Discussions

A PROPOSED NEW RELATION FOR CUMULATIVE FATIGUE DAMAGE IN BENDING*

By S. S. Manson, A. J. Nachtigall, and J. C. Freche[1]

Synopsis

A procedure is proposed for determining the remaining fatigue life of specimens subjected to a prior history of cyclic bending stresses. The method can be applied either graphically or analytically, the only requirement being a knowledge of a characteristic constant which is readily obtainable by laboratory tests or which can be estimated.

The fatigue behavior of electric-furnace-melted SAE 4130 steel was investigated in rotating-bending fatigue after it was subjected to a prestress at 100,000 psi for different cyclic histories. This behavior corroborated the hypothesis proposed, which is based upon the assumption that lines representing the S - log N relationship of material prestressed varying amounts will intersect the S - log N line of the original material near a common point.

Comparison of the proposed procedure with that of Palmgren-Miner indicates that the latter method is a special case of that described herein. The order-dependence effect of load application upon fatigue life, as well as the reduction in endurance limit which occurs when a material is prestressed, is accounted for in the proposed procedure. Neither of these factors is considered in the Palmgren-Miner method.

The proposed procedure appears to afford a useful design tool, although more experimental verification is required with other materials, other stress histories, and other types of fatigue tests before the usefulness of this approach can be clearly established.

The impossibility of duplicating in the laboratory the infinite variety of load spectra encountered in engineering practice, and thereby predicting fatigue life in advance of service, has led to the advancement of various cumulative fatigue damage theories **(1–8)**.[2] Many of these **(1–5)** methods consider fatigue damage to be stress-dependent and require experimental procedures to evaluate the stress dependence. As a consequence, these methods are often too complex to be readily applied by the designer. There are also available to designers some readily applicable methods for predicting fatigue life, such as those of references **(6,7,8)**. These can provide useful approximations of the remaining fatigue life after a prior load history has been imposed; however, the simpler methods often display limited accuracy.

The most widely known of the simpler procedures, that of Palmgren-Miner **(6–7)**, is a summation-of-cycle ratio hypothesis. As has been pointed out **(9)**, a number of investigations have shown results of specific tests to be incompatible with a simple summation of cycle ratios. The fact that this method does not account for the order-dependence effect of load application upon fatigue life nor consider the reduction in endurance limit which occurs when a material is prestressed, contributes to the introduction of errors in predicting fatigue life when it is used. Despite these drawbacks the method of cycle ratio summation has the overriding advantage of simplicity. It would be desirable therefore to devise means whereby this basic simplicity is retained and at the same time account for the deviations from observed behavior which occur when a summation of cycle ratios is employed to predict fatigue life.

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* Presented at the Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] Lewis Research Center, National Aeronautics and Space Administration, Cleveland, Ohio.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

In order to gain insight into the simplest types of modifications that can be made, it is desirable to consider the elementary case in which a series of specimens are all first subjected to the same known prior history and then used to determine the "remaining" S - log N curve. Examination of the Palmgren-Miner method indicates that it predicts that S - log N curves obtained for materials with a prior fatigue history will be parallel to the S - log N curve of the original material. If, however, as predicated by Henry (8), the prior damage consists essentially of the introduction of minute cracks which produce an effective stress concentration, then it will not be expected that the remaining S - log N curve will be parallel to the curve for the original material.

It has generally been observed that the effect on fatigue life of stress concentrations introduced into a material by prior stressing is less pronounced in the region of high stress (low cyclic life) than in the region of low stress (high cyclic life). Therefore, it might be expected that the S - log N line for a prestressed material would tend to converge toward the S - log N line of the original material in the high stress region rather than remain parallel to it. The point of intersection between the original S - log N line and that of the prestressed material then becomes an important parameter characteristic.

If the hypothesis of convergence to a common point for all prior histories is assumed to be valid, an expression for predicting fatigue life can be written which differs from that of Miner but which retains much of the simplicity of the basic summation-of-cycle ratio hypothesis. The effect of prior history then becomes expressible in terms of the coordinates of the common point of intersection, this point being a function only of the material, and perhaps of the type of fatigue test used in the evaluation. Such an expression would have the advantage of incorporating the effect of order-dependence upon fatigue life. An additional expression can be written for calculating the amount of reduction in endurance limit which occurs when a material is prestressed. Limited verification of this convergence concept has been obtained by Bennett (10) in a series of fatigue studies with X4130 steel, but much experimental work is required in order to provide adequate substantiation of this concept. In addition it is necessary to derive the necessary analytical expressions expressing this geometrical relation.

This paper presents the results of a new experimental investigation conducted with heat-treated SAE 4130 steel specimens which were subjected to bending fatigue according to a program of prior stressing. Since these results tended to confirm the convergence of S - log N lines at a common point, a method based on this concept for predicting the remaining fatigue life of a material subjected to a complex load history is also proposed. This method is shown to lend itself equally well to a graphical or analytical procedure, and the results of its application to a few simple load histories provide an indication of its usefulness.

It should be noted that the proposed procedure is based upon observed experimental behavior and not upon fundamental fatigue mechanisms. As such it represents an engineering and not a fundamental approach to the problem of cumulative fatigue damage. Although the procedure is intended for application to complex as well as simple prior load histories, the scope of the present investigation is limited to a relatively simple prestress regime in bending fatigue. Its usefulness for other types of fatigue tests under conditions of complex prior load histories as well as with other materials remains to be determined.

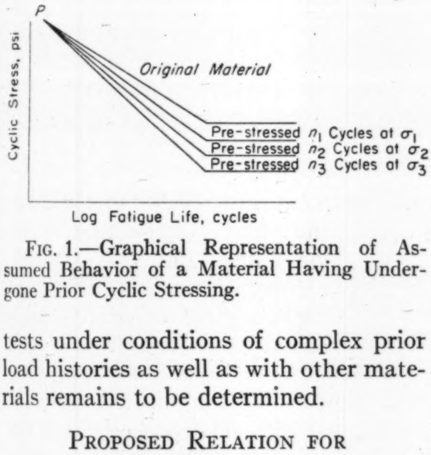

FIG. 1.—Graphical Representation of Assumed Behavior of a Material Having Undergone Prior Cyclic Stressing.

PROPOSED RELATION FOR CUMULATIVE FATIGUE

The proposed procedure can be applied graphically or analytically. The graphical application provides a visual explanation of the concepts involved and will be considered first.

Graphical Application of Proposed Relation:

Figure 1 provides a graphical representation of the assumed behavior of a material that has undergone prior stressing in terms of S - log N lines plotted on a semilog scale. The S - log N lines of the variously prestressed material are shown to be straight and to intersect the original material S - log N line at a common point, P. The value of N at this intersection point is then assumed to be a constant associated with the material and the condition of testing which can be determined by laboratory evaluation or by approximation. The assumption of linearity would appear to be sufficiently valid for most ferrous materials. Although the assumption is not rigorously correct, it is possible to approximate the S - log N curves by straight lines without introducing a high degree of error with many materials. Observations show that a similar convergence and a similar approximation of linearity is obtained on log S - log N type plots, although the assumed point of convergence is different. Additional comments regarding this latter method of plotting fatigue data will be made later.

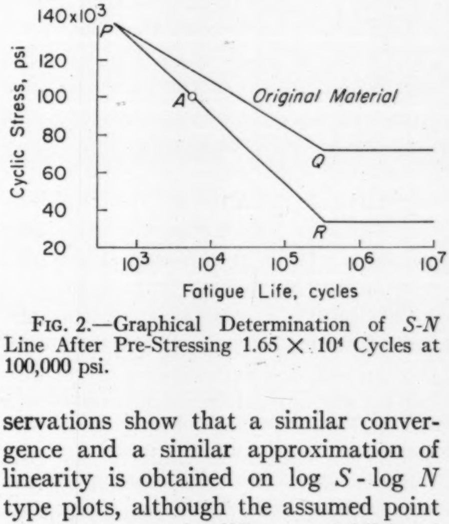

FIG. 2.—Graphical Determination of S-N Line After Pre-Stressing 1.65×10^4 Cycles at 100,000 psi.

Once the common intersection point is chosen, it then becomes a simple matter to predict remaining fatigue life after any given prestress history has been applied. This is illustrated for a simple case in Fig. 2. Some material is here shown to have been prestressed to 75 per cent of its expected life of 2.2×10^4 cycles at a 100,000-psi stress level. The life remaining to the material at this stress level obviously is 25 per cent of 2.2×10^4

cycles, or 5.5 × 10^3 cycles. This point may then be plotted as point A in Fig. 2. A line drawn through this point and the point P represents the remaining fatigue life of a material, so prestressed, over the complete range of stress levels. This procedure can of course be extended to additional prestress conditions, if it is borne in mind that for a second prestress condition, remaining life must be determined from the S - log N line PR which describes the behavior of the material after the first prestress condition.

In Fig. 1 it is assumed that the cyclic life to the knee of the S - log N lines (endurance limits) of the variously prestressed materials will be constant and that this value is the same as that which obtains at the endurance limit of the original material. Although the data show that such an assumption is only very approximate, its use makes it possible without further testing to take into account those portions of a prestress history obtained at low stress levels. In any graphical determination of remaining fatigue life where a portion of the stress history is imposed at a stress level below the endurance limit of the prestressed material, this portion of the stress history obviously should not be included in the cycle ratio summation. Under such circumstances, the fatigue life probably is not adversely affected since the sample is being stressed in a region of infinite life.

Analytical Application of Proposed Relation:

If it is assumed that the S - log N lines of the variously stressed materials are straight and intersect at a common point as described previously, it is possible to derive equations expressing cumulative fatigue damage by employing simple geometric considerations. The detailed derivation procedure is presented in Appendix I. The equation describing the simplest case, that of a single prestress condition, is:

$$n_{r,2} = N_2\left(1 - \frac{n_1}{N_1}\right)^{\frac{\log N_2/N_P}{\log N_1/N_P}} \quad \ldots\ldots(1)$$

The equation for the general case of any number of prestress conditions is:

$$\frac{n_{r,i+1}}{N_{i+1}} = \left\{\cdots\left\{\left[\left(1 - \frac{n_1}{N_1}\right)^{\frac{\log N_2/N_P}{\log N_1/N_P}} - \frac{n_2}{N_2}\right]^{\frac{\log N_3/N_P}{\log N_2/N_P}} - \frac{n_3}{N_3}\right\}^{\frac{\log N_4/N_P}{\log N_3/N_P}} \cdots - \frac{n_i}{N_i}\right\}^{\frac{\log (N_{i+1}/N_P)}{\log (N_i/N_P)}} \quad \ldots(2)$$

where:

n = number of cycles applied,

$n_{r,i+1}$ = number of cycles of life remaining after i number of prestresses (that is, number of cycles of life remaining at the $i + 1$ stress level),

N = number of cycles to failure on original material S - log N line,

N_P = number of cycles to failure at the "best" intersection of S - log N lines, and

N_{i+1} = number of cycles of life according to original material S - log N line at $i + 1$ stress level.

Although the equations appear at first glance to be complicated, they can be applied with relative ease. They are markedly similar to the equations which describe the Palmgren-Miner hypothesis. When the term, N_P, becomes infinity, the exponential ratios can be considered as unity and the equations revert to the form of Miner's equations. These latter equations accounted for the effect of a given portion of a complex prior load history by adding the cycle ratio associated with this portion to the summation of the cycle ratios describing the remainder of the prestress history. The present method alters this process only to the extent that before the cycle ratio of a given prestress portion is added, the systematically modified cycle ratio expressing the preceding part of the load history is raised to a known exponent. The order-dependency effect of load application is taken into account, in contrast to the order independence of the Palmgren-Miner procedure. Once the constant, N_P, has been determined the remaining fatigue life after any number of prestress conditions may be readily calculated.

Determination of Best Intersection of S - log N lines:

The evaluation of the constant N_P represents the only experimental procedure required for evaluating the above equations. Since the S - log N lines of the variously prestressed material are assumed to intersect in a common region, it becomes necessary to establish only the S - log N line for one prestress condition. The original material S - log N line is usually available to designers. When experimental facilities are not available, an approximation of the constant, N_P, can be made. By employing a commonly used approximation (**11**), it appears that a reasonable approximation of the value of N_P is possible for steels. Reference (**11**) shows that a material that has received prior surface treatment, which can be regarded as surface damage, provides a series of straight converging lines on a log stress versus log cycle life type plot. These intersect the nontreated material line at approximately 1000 cycles. The data obtained herein when plotted as log stress versus log cycle life also show the best intersection point of the prestressed material lines and the original material line to be at 1000 cycles. The cyclic life at the "best" intersection of the S-N lines is higher when the data of this investigation are plotted as log stress versus log cycle life, than when a S - log N (semi-log) type plot is used. However, the effect upon predicted values of remaining fatigue life by the proposed procedure is not marked, whether the constant, N_P, chosen is 1000 cycles or as low as 100 cycles.

Effect of Operation Below Endurance Limit of Original Material:

In summing up the cycle ratios that comprise a given prestress history, the evaluation of the equations cited poses no difficulties when the stress levels involved lie above the endurance limit of the original (not prestressed) material. When the stress level is below this limit the question arises whether or not this particular portion of the prestress history should be included. To evaluate this consideration properly requires a knowledge of the location of the new endurance limit of the prestressed material. If the stress level in question lies below this endurance limit, it should not be included, since the fatigue life is probably not adversely affected by this particular portion of its prestress history. Any improvement in material properties that might result from such prestressing is not accounted for by the proposed theory. It may also be noted that, although considerable evidence exists which shows the improvement in fatigue properties by understressing to be a real phenomenon, other investigators (**12**) have indicated that such an improvement can be interpreted as a statistical phenomenon based on selectivity. If the stress level in question lies between the endurance limits of the prestressed and original material, this portion of the prestress history should be included in the prestress summation. This is done by extending the inclined portion of the original material S - log N line, reading off the expected life at that stress level, and using this value to determine the cycle ratio, n/N, for inclusion in Eq 2. The reason for this procedure is apparent from a consideration of the derivation of the equations for calculating the endurance limits.

The endurance limit of a prestressed material can be calculated fairly readily if the assumption previously cited (that is, the cyclic life to the knee of the S - log N lines for the variously prestressed materials is constant and this value is the same as that which obtains at the endurance limit of the original material) is made. This is done in Appendix II where equations for predicting the endurance limits of prestressed materials are derived. For the simplest case of a single prestress the equation is:

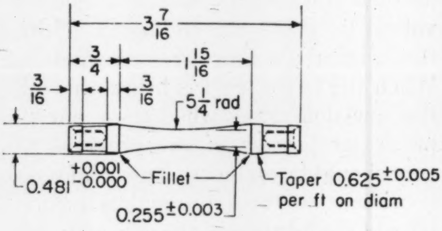

FIG. 3.—Fatigue Specimen.

$$\sigma_{E,1} = \sigma_P - \frac{(\sigma_P - \sigma_{E,O})}{1 + \log\left(1 - \frac{n_1}{N_1}\right)^{\frac{1}{\log N_1/N_P}}} \quad (3)$$

For several prestress cycle ratios, a more general equation can be written:

$$\sigma_{E,i} = \sigma_P - \frac{(\sigma_P - \sigma_{E,O})}{1 + \log\left\{\left\{\cdots\left\{\left[\left(1 - \frac{n_1}{N_1}\right)^{\frac{\log N_2/N_P}{\log N_1/N_P}} - \frac{n_2}{N_2}\right]^{\frac{\log N_3/N_P}{\log N_2/N_P}} - \frac{n_3}{N_3}\right\}^{\frac{\log N_4/N_P}{\log N_3/N_P}} \cdots - \frac{n_{i-1}}{N_{i-1}}\right\}^{\frac{\log N_i/N_P}{\log N_{i-1}/N_P}} - \frac{n_i}{N_i}\right\}^{\frac{1}{\log N_i/N_P}}} \quad (4)$$

where:

σ = applied cyclic stress,

$\sigma_{E,O}$ = stress at endurance limit of original material,

Subscripts E, 1 E, i = endurance limit after first prestress, and any specified prestress, respectively.

Both equations are expressed in terms of similar parameters to those used in Eqs 1 and 2; cycle ratios, the constant, N_P, and the term $\sigma_{E,O}$, the initial endurance limit.

EXPERIMENTAL PROCEDURE

Materials:

Electric-furnace-melted, heat-treated SAE 4130 steel specimens were subjected to bending fatigue in R. R. Moore rotating-beam fatigue machines. A sketch of the specimens used is shown in Fig. 3. The material was solution treated for $\frac{1}{2}$ hr at 1700 F, water quenched, annealed for $\frac{1}{2}$ hr at 1200 F, and air-cooled to room temperature. After heat treatment, the test specimens were machined. The test section of each was machined to within 0.003 in. of the final dimension shown in Fig. 3 and machine polished with abrasive cloth of three grit sizes (320, 400, and 500) to attain the final dimension. Approximately 300 specimens were employed in this investigation.

Several inspection procedures were employed during various stages of test specimen preparation. Specimen blanks were first subjected to an ultrasonic inspection to sort out those with gross internal imperfections. After final polishing the specimens were subjected to an oil emulsion inspection (Zyglo). The final inspection consisted of a macroscopic examination using a binocular microscope (×20). This inspection was made over $\frac{1}{4}$ in. of the test section. All specimens containing more than 30 pits in this region were rejected because preliminary operation indicated that an unusually high incidence of premature failure resulted when such samples were used. After final inspection all specimens were coated with a thin protective oil film to avoid atmospheric corrosion.

Tests:

Tests were conducted at 5000 rpm (±30 rpm). A specimen run-out no greater than 0.001 in. full indicator reading was permitted upon installation into the fatigue machines. To determine the inclined portion of the S - log N line of the original material, three groups of specimens were run to failure: one at 120,000 psi, one at 100,000 psi, and one at 85,000 psi. To determine the S - log N lines for various prestress conditions, specimens were first selected for operation to failure at a particular stress level. Then each specimen was operated for a prescribed number of cycles at a specified stress to achieve the desired prestress condition. A variety of single prestress conditions were considered at 100,000 psi. For this stress, 1.7×10^4, 1.4×10^4, and 1.0×10^4 prestress cycles were applied. Once the prestress operation was completed, the load of the machine was changed and the test continued to failure.

The endurance limit was determined in several instances. The number of specimens available for these determinations was limited. The method consisted of conducting tests at close intervals (1000 psi) in the assumed region of the endurance limit. For the prestressed material S - log N lines, the samples were first prestressed, and those which did not fail in prestressing were subsequently used to determine the endurance limit.

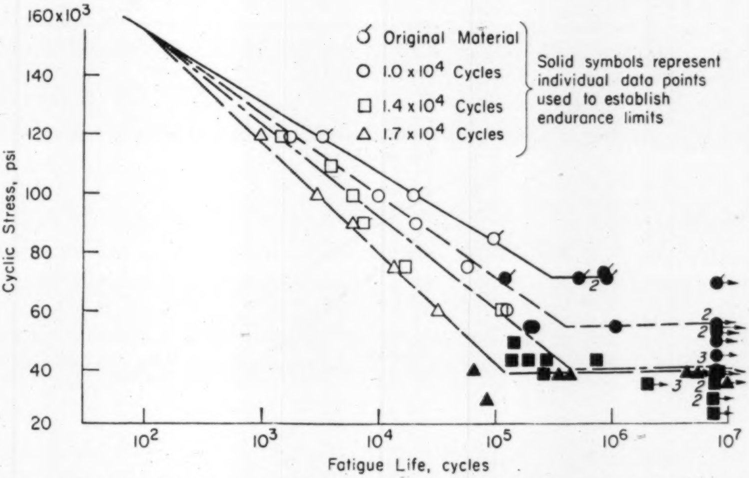

FIG. 4.—Comparison of S - log N Lines for Original and Pre-Stressed SAE 4130 Heat Treated Material. Cyclic Pre-Stress Conditions were at 100,000 psi. Median points shown in all cases.

EXPERIMENTAL RESULTS

Approximately eleven data points were obtained at most of the stress levels considered in arriving at the S - log N lines. Figure 4 shows only the median data points. Use of the median was considered to be more significant than use of the mean because of the unsymmetrical distribution of fatigue life values. Also, since prestress failures do occur in some instances, the average or mean value of life would be affected out of proportion to the significance of a prestress failure.

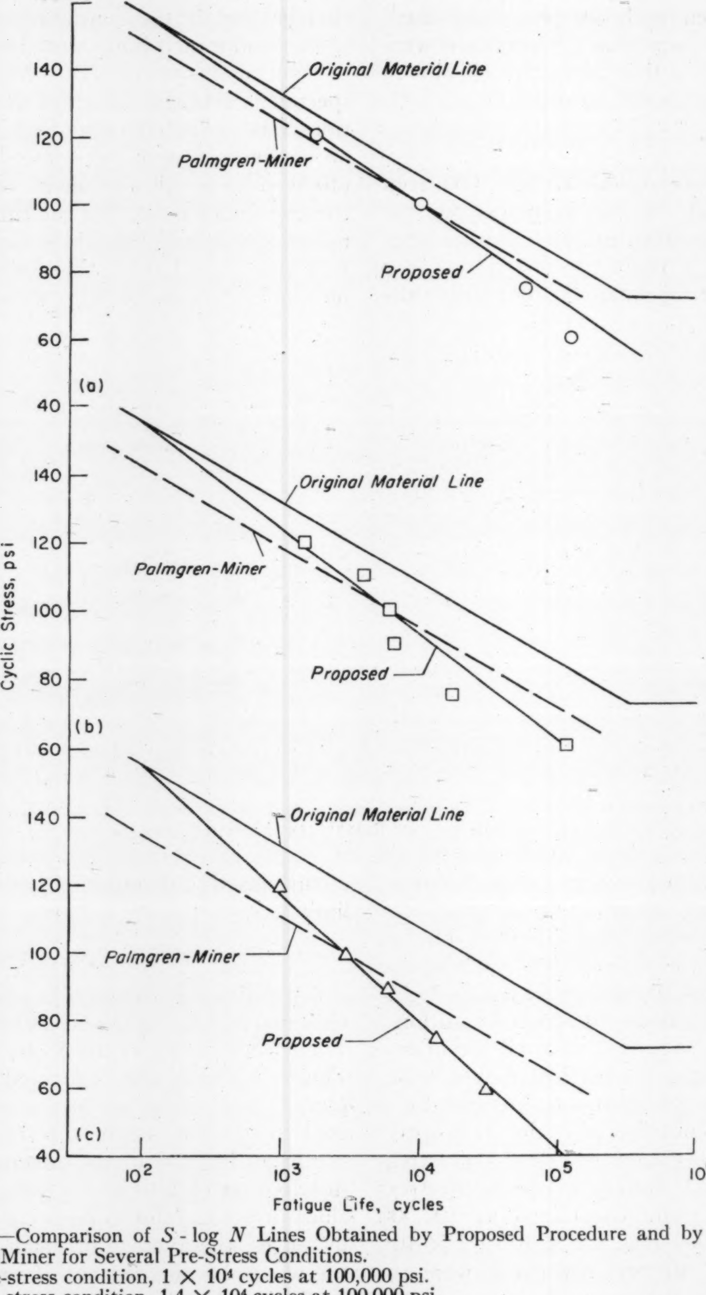

FIG. 5.—Comparison of S - log N Lines Obtained by Proposed Procedure and by Method of Palmgren-Miner for Several Pre-Stress Conditions.

(*a*) Pre-stress condition, 1 × 10^4 cycles at 100,000 psi.
(*b*) Pre-stress condition, 1.4 × 10^4 cycles at 100,000 psi.
(*c*) Pre-stress condition, 1.7 × 10^4 cycles at 100,000 psi.

Use of the median is recommended in reference (13) as well.

The lines drawn through the data points on Fig. 4 converge and intersect at 100 cycles and a stress of 156,000 psi. The common intersection point lies upon the original material S - log N line. A gradually steeper slope is evident as the number of prestress cycles is increased. The increasing slope indicates a decreasing remaining fatigue life as might be expected.

Also shown in Fig. 4 are the data points obtained to locate the endurance limits of the original as well as the prestressed material. These are shown blocked in. Only limited data were obtained to establish these endurance limits and their location is therefore only approximately defined. However, the data do provide some basis for the assumption that the endurance limits of the variously prestressed materials fall in line at a constant value of cycle life, used in the derivation of Appendix II. It may be that additional determinations will indicate that the prestressed material endurance limits do not line up as postulated, but rather that they occur at successively smaller values of cycle life as suggested by the 1.7 × 10^4 cycle prestress data. In that case, it would still be possible to derive mathematically an expression for predicting such endurance limits, using a more probable assumption regarding the locations of the endurance limits.

A comparison of the S - log N lines obtained by the proposed procedure and by the method of Palmgren-Miner is shown in Fig. 5. The deviation of the prestress data from the prestressed material S - log N lines defined by Palmgren-Miner is also shown in this figure. The 1.7 × 10^4 cycle prestress data illustrate rather well the converging effect, which is the major premise of the proposed procedure. The data for the other two prestress conditions are not nearly as conclusive. This emphasizes the fact that additional verification of the proposed procedure is required for other prestress histories as well as other materials.

DISCUSSION

If predictions of remaining fatigue life were made for the prestress conditions considered in this investigation by the proposed procedure and by that of Palmgren-Miner, a considerable difference would be noted between the results obtained by each method. This is apparent from an examination of Fig. 5. The deviation of the data points from the line of Palmgren-Miner is obviously greater than from the S - log N line describing the proposed procedure. The 1.7 × 10^4 cycle prestress condition shows this most markedly.

Of course it should be recognized that Miner (7) limits the application of his method to aluminum and to the region above the endurance limit of the original (nonprestressed) material. Miner's method has, however, been applied by designers to other materials. In such applications it generally affords a simple means of indicating an order of magnitude type of result, when fatigue life predictions are confined to the region above the original material endurance limit. When Miner's method is applied to regions below the endurance limit of the original material, after a prior stress history has been imposed above this endurance limit, predictions of remaining fatigue life can lead to considerable error. For example, at all stresses below the endurance limit of the original material, infinite life would be predicted by Miner's method since it does not provide for the decrease which occurs in the endurance limit of prestressed material relative to that of the original material. That infinite life is not obtained experimentally is apparent from Fig. 4, which clearly shows a reduction in the endurance limits of variously prestressed materials. It should be noted, however, that the procedure suggested herein for calculating the new endurance limit of prestressed material can also be applied in conjunction with Miner's summation-of-cycle ratio process. If this is done, the S - log N line of the prestressed material as predicted by Miner's hypothesis can be extended to a point which falls on a line of constant cyclic life, along which the endurance limits of the original and the prestressed material are assumed to fall. As a consequence, the usefulness of Miner's method could be considerably improved.

The method of Henry (**8**) has also been mentioned as one which was readily applicable for predicting fatigue life. Henry suggests that prior stressing produces an effective stress concentration in a material which persists throughout subsequent operation at different stress levels. The application of this concept in predicting remaining fatigue life of materials which have been prestressed results in a series of lines on semilogarithmic paper which diverge from the line expressing the S - log N relationship of the original material (not prestressed) at the higher stresses. It should be recognized that other investigators (**14,15**) have, in a few instances, shown a somewhat similar divergence (at the higher stresses) of the S - log N lines obtained for notched and unnotched materials. In some of these cases although divergence occurs at extremely high stresses (above tensile strength of material), the S - log N lines nevertheless intersect near the tensile strength of the materials in question. In others, there does not appear to be an intersection, although a slight convergence is noted at the lower stresses. These results merely serve to emphasize the need for additional experimental fatigue investigations. It remains to be seen whether the proposed procedure can be applied in those instances where the S - log N lines converge at lower stresses (higher cyclic lives). Many more conditions of prior stressing as well as other materials must be investigated in order to make conclusive judgments as to the validity of the proposed procedure, even for ferrous materials. Additional tests are also required before its applicability to cumulative damage engendered by fatigue tests other than alternating bending can be ascertained. The significance of the constant N_P should also be determined in order to find out whether it is related primarily to the type of material, the type of tests employed, or the type of specimens employed (notched or unnotched). Despite a lack of understanding regarding these factors, the data presented together with those already obtained by Bennett (**10**), suggest that the procedure proposed herein gives promise of becoming a useful design tool.

Acknowledgment:

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APPENDIX I

DERIVATION OF EQUATIONS FOR EXPRESSING CUMULATIVE FATIGUE DAMAGE

In deriving an expression for remaining fatigue life after prestressing, the simplest case to be considered is that for the condition of a single prestress. Utilizing the nomenclature of Fig. 6, it is apparent that the ratio of distances BC to EF equals that of AC to

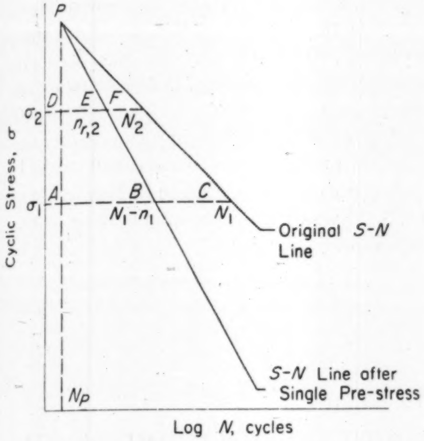
P
D
E
F
σ_2
$n_{r,2}$
N_2
A
B
C
σ_1
N_1-n_1
N_1
Original S-N Line
S-N Line after Single Pre-stress
N_P
Cyclic Stress, σ
Log N, cycles

FIG. 6.—General Location of S - log N Lines for Original Material and Material Subjected to a Single Pre-Stress Condition.

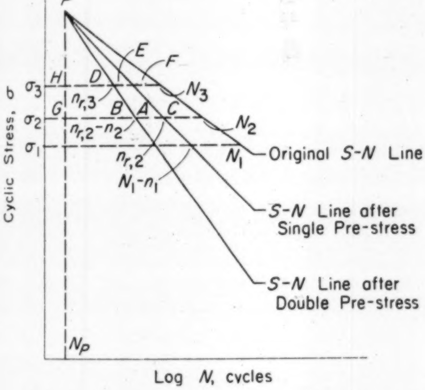
P
E
F
H
D
σ_3
N_3
$n_{r,3}$
G
B
A
C
σ_2
N_2
$n_{r,2}-n_2$
σ_1
$n_{r,2}$
N_1
N_1-n_1
Original S-N Line
S-N Line after Single Pre-stress
S-N Line after Double Pre-stress
N_P
Cyclic Stress, σ
Log N, cycles

FIG. 7.—General Location of S - log N Lines for Original Material and Materials Subjected to a Single and Double Pre-Stress Condition.

DF. Expressed in terms of the variables plotted, this ratio becomes:

$$\frac{\log N_1 - \log (N_1 - n_1)}{\log N_2 - \log n_{r,2}} = \frac{\log N_1 - \log N_P}{\log N_2 - \log N_P} \quad (A1)$$

By a series of algebraic manipulations such as the following, the final simplified form of the equation may be obtained:

$$\frac{\log \dfrac{N_1 - n_1}{N_1}}{\log \dfrac{n_{r,2}}{N_2}} = \frac{\log \dfrac{N_1}{N_P}}{\log \dfrac{N_2}{N_P}} \quad (A2)$$

$$\log \frac{n_{r,2}}{N_2} = \log \left(1 - \frac{n_1}{N_1}\right) \frac{\log \dfrac{N_2}{N_P}}{\log \dfrac{N_1}{N_P}} \quad (A3)$$

$$\frac{n_{r,2}}{N_2} = \left(1 - \frac{n_1}{N_1}\right)^{\log \frac{N_2}{N_P} / \log \frac{N_1}{N_P}} \quad (A4)$$

or

$$n_{r,2} = N_2 \left(1 - \frac{n_1}{N_1}\right)^{\log \frac{N_2}{N_P} / \log \frac{N_1}{N_P}} \quad (A5)$$

which expresses the number of cycles of life remaining, $n_{r,2}$, at stress, σ_2, after the material has been prestressed, n_1, cycles at stress, σ_1.

The equation for the conditions of two or more prestresses is merely an extension of Eq ($A5$) and may readily be obtained by compounding the foregoing procedure. Utilizing the nomenclature of Fig. 7 it is seen from the relation between similar triangles that the distance ratio BC to DF equals that of CG to FH. In terms of cycle ratios this can be written as

$$\frac{\log N_2 - \log (n_{r,2} - n_2)}{\log N_3 - \log n_{r,3}} = \frac{\log N_2 - \log N_P}{\log N_3 - \log N_P} \quad (A6)$$

Simplifying and transposing gives:

$$\log \left(\frac{n_{r,2}}{N_2} - \frac{n_2}{N_2}\right) = \log \frac{n_{r,3}}{N_3} \cdot \frac{\log \dfrac{N_2}{N_P}}{\log \dfrac{N_3}{N_P}} \quad (A7)$$

Substituting Eq ($A4$) into Eq ($A7$) gives:

$$\log \left[\left(1 - \frac{n_1}{N_1}\right)^{\frac{\log N_2/N_P}{\log N_1/N_P}} - \frac{n_2}{N_2}\right] = \log \frac{n_{r,3}}{N_3} \cdot \frac{\log \dfrac{N_2}{N_P}}{\log \dfrac{N_3}{N_P}} \quad (A8)$$

This equation can be rewritten in the form of Eq (*A*5) as:

$$\frac{n_{r,3}}{N_3} = \left[\left(1 - \frac{n_1}{N_1}\right)^{\frac{\log N_2/N_P}{\log N_1/N_P}} - \frac{n_2}{N_2}\right]^{\frac{\log N_3/N_P}{\log N_2/N_P}} \quad ..(A9)$$

and expresses the fatigue life remaining in terms of the ratio, $n_{r,3}/N_3$, at a third stress level, σ_3, after the material has been prestressed for, n_1, and n_2, cycles at two stresses, σ_1, and σ_2.

A general expression for any greater number of prestress conditions can now be deduced from consideration of Eqs (*A*4) and (*A*9). This general expression may be written as follows:

$$\frac{n_{r,i+1}}{N_{i+1}} = \left\{\cdots\left\{\left[\left(1 - \frac{n_1}{N_1}\right)^{\frac{\log N_2/N_P}{\log N_1/N_P}} - \frac{n_2}{N_2}\right]^{\frac{\log N_3/N_P}{\log N_2/N_P}} - \frac{n_3}{N_3}\right\}^{\frac{\log N_4/N_P}{\log N_3/N_P}} \cdots - \frac{n_i}{N_i}\right\}^{\frac{\log N_{i+1}/N_P}{\log N_i/N_P}} \quad ..(A10)$$

APPENDIX II

DERIVATION OF EQUATIONS TO PREDICT ENDURANCE LIMIT STRESS

Expression for Condition of Single Prestress:

The derivation of the endurance limit stress after prestressing for a known number of cycles is based primarily on the assumption that the horizontal portion of the *S* - log *N* line for any prestress condition will begin at the same number of cycles, N_E, as it does for the original material *S* - log *N* line. From the geometry of similar triangles *PBC* and *PDE* in Fig. 8 the following proportionality can be written:

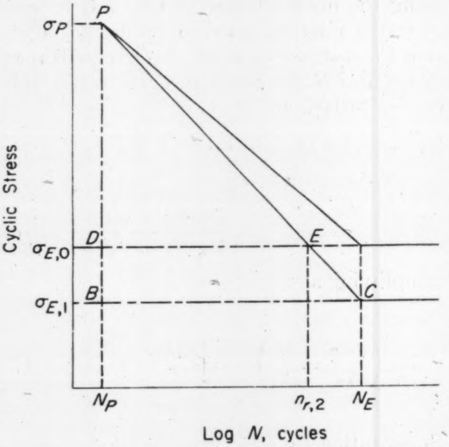

FIG. 8.—Illustration of Original Material and Single Pre-Stress Endurance Lines Used to Derive Equations for Predicting Endurance Limit Stress.

$$\frac{\sigma_P - \sigma_{E,1}}{\sigma_P - \sigma_{E,O}} = \frac{\log N_E - \log N_P}{\log n_{r,2} - \log N_P} \quad ..(B1)$$

Letting $N_2 = N_E$ in Eq (*A*5) from Appendix I:

$$\log n_{r,2} = \log N_E + \frac{(\log N_E - \log N_P)}{(\log N_1 - \log N_P)} \log\left(1 - \frac{n_1}{N_1}\right) \quad ..(B2)$$

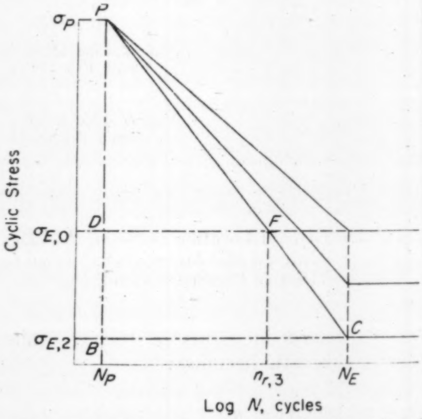

FIG. 9.—Illustration of Original Material and Double Pre-Stress Endurance Lines Used to Determine Equations for Predicting Endurance Limit Stress.

Substituting this expression into Eq (*B*1) and cancelling the common terms (log N_E − log N_P) from numerator and denominator yield:

$$\frac{\sigma_P - \sigma_{E,1}}{\sigma_P - \sigma_{E,O}} = \frac{1}{1 + \dfrac{\log\left(1 - \dfrac{n_1}{N_1}\right)}{\log \dfrac{N_1}{N_P}}} \quad ..(B3)$$

Solving for the endurance limit after a single prestress yields:

$$\sigma_{E,1} = \sigma_P - \frac{(\sigma_P - \sigma_{E,O})}{1 + \log\left(1 - \dfrac{n_1}{N_1}\right)^{\frac{1}{\log N_1/N_P}}} \quad ..(B4)$$

Expression for Condition of Two or More Prestresses:

From the geometry of similar triangles *PBC* and *PDF* in Fig. 9 the following proportionality can be written:

$$\frac{\sigma_P - \sigma_{E,2}}{\sigma_P - \sigma_{E,O}} = \frac{\log N_E - \log N_P}{\log n_{r,3} - \log N_P} \quad ...(B5)$$

If it is assumed that $N_3 = N_E$ in Eq ($A9$) of Appendix I, then,

$$\log n_{r,3} = \log N_E + \frac{(\log N_E - \log N_P)}{(\log N_2 - \log N_P)} \cdot \log\left[\left(1 - \frac{n_1}{N_1}\right)^{\frac{\log N_2/N_P}{\log N_1/N_P}} - \frac{n_2}{N_2}\right] \quad ..(B6)$$

Substituting this expression into Eq ($B5$) and cancelling the common terms ($\log N_E - \log N_P$) from numerator and denominator yield,

$$\frac{\sigma_P - \sigma_{E,2}}{\sigma_P - \sigma_{E,O}} = \frac{1}{1 + \dfrac{\log\left[\left(1 - \dfrac{n_1}{N_1}\right)^{\frac{\log N_2/N_P}{\log N_1/N_P}} - \dfrac{n_2}{N_2}\right]}{\log \dfrac{N_2}{N_P}}} \quad .(B7)$$

Solving for the endurance limit $\sigma_{E,2}$ after two prestress levels of n_1 and n_2 cycles gives,

$$\sigma_{E,2} = \sigma_P - \frac{(\sigma_P - \sigma_{E,0})}{1 + \log\left[\left(1 - \dfrac{n_1}{N_1}\right)^{\frac{\log N_2/N_P}{\log N_1/N_P}} - \dfrac{n_2}{N_2}\right]^{\frac{1}{\log N_2/N_P}}} \quad ....(B8)$$

A general expression for any greater number of prestress conditions can now be deduced from consideration of Eqs ($B4$) and ($B8$). This general expression can be written as follows:

$$\sigma_{E,i} = \sigma_P - \frac{(\sigma_P - \sigma_{E,O})}{1 + \log\left\{\left\{\cdots\left\{\left[\left(1 - \dfrac{n_1}{N_1}\right)^{\frac{\log N_2/N_P}{\log N_1/N_P}} - \dfrac{n_2}{N_2}\right]^{\frac{\log N_3/N_P}{\log N_2/N_P}} - \dfrac{n_3}{N_3}\right\}^{\frac{\log N_4/N_P}{\log N_3/N_P}} \cdots - \dfrac{n_{i-1}}{N_{i-1}}\right\}^{\frac{\log N_i/N_P}{\log N_{i-1}/N_P}} - \dfrac{n_i}{N_i}\right\}^{\frac{1}{\log N_i/N_P}}} \quad ..(B9)$$

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DISCUSSION

MR. R. L. BENEDICTO[1] (*presented in written form*).—The proposed method given in the paper has been used to calculate predicted lives for 17 test groups totaling 70 specimens, as reported in NACA TN 4284. The tests were performed on 4130 steel heat treated to ultimate tensile strength of 133,800 psi, using polished specimens in rotating bending. Although TN 4284 covers tests at room temperature and at elevated temperatures, the room-temperature tests were the only ones used to check the proposed method. A value of $N_p = 1000$ cycles was used, as suggested in the authors' paper.

The accompanying Tables I and II give the predicted lives by the Miner-Palmgren method, Henry's method, and the proposed method along with the test results. It can be seen that the proposed method always gives an answer intermediate between the answers obtained by using Miner's and Henry's methods. In general, Henry's method seems to give answers closer to the test results, with the life predictions by Henry's method falling within the test scatter range more often and the per cent deviation from the median test values being less than for the other two methods.

It should also be noted that neither Henry's method nor the proposed method can be used to predict the fatigue life of an actual structure in service when the loads are applied in a random manner. The major usefulness of the methods seems to be in providing understanding of results obtained in laboratory tests.

MR. J. C. FRECHE (*author*).—The authors wish to thank Mr. Benedicto for bringing up a subject which affords an opportunity to emphasize the differences between the proposed procedure and those of Miner and Henry. It was, in

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TABLE I.—NACA TN 4284 TEST DATA (ROOM TEMPERATURE).
Comparison of Test Lives with Various Predictions.

Test Data							Predicted $\Sigma n/N$		
S_1, psi	S_2, psi	n_1/N_1	$\Sigma n/N$				Miner	Henry	ASTM Paper
			Min	Max	Avg	Median			
80 000	88 000	0.250	0.999	1.268	1.134	1.134	1.000	1.160	1.037
80 000	88 000	0.500	1.434	1.561	1.481	1.466	1.000	1.271	1.061
80 000	88 000	0.750	1.114	1.544	1.374	1.420	1.000	1.279	1.065
88 000	80 000	0.250	0.619	0.817	0.740	0.767	1.000	0.718	0.958
88 000	80 000	0.500	0.631	0.957	0.829	0.870	1.000	0.733	0.935
88 000	80 000	0.750	0.873	1.463	1.107	1.046	1.000	0.840	0.940
82 000	94 000	0.250	0.837	1.198	0.940	0.862	1.000	1.150	1.059
82 000	94 000	0.500	0.792	1.267	1.031	0.982	1.000	1.246	1.101
82 000	94 000	0.750	0.954	1.176	1.080	1.094	1.000	1.247	1.111
94 000	82 000	0.250	0.504	0.883	0.652	0.611	1.000	0.750	0.926
94 000	82 000	0.500	0.539	0.752	0.655	0.665	1.000	0.753	0.890
94 000	82 000	0.750	0.760	1.025	0.870	0.848	1.000	0.851	0.902

NOTE.—All tests on 4130 steel heat treated to MIL-S-6758, condition F5 (ultimate tensile strength, 133.8 ksi). Smooth specimens in rotating-beam fatigue.

Cycled at S_1 to give required value of n_1/N_1, and then cycled to failure at S_2. Four specimens tested in each group.

TABLE II.—NACA TN 4284 TEST DATA (ROOM TEMPERATURE).
3-Step and 5-Step Test Results and Predictions.

TEST PARAMETERS

Test	S_1, psi	S_2, psi	S_3, psi	S_4, psi	S_5, psi	n_1/N_1	n_2/N_2	n_3/N_3	n_4/N_4
3A......	82 000	88 000	94 000	...	...	0.300	0.300	to failure	...
3B......	94 000	88 000	82 000	...	...	0.300	0.300	to failure	...
5A......	82 000	85 000	88 000	91 000	94 000	0.200	0.200	0.200	0.200
5B......	94 000	91 000	88 000	85 000	82 000	0.200	0.200	0.200	0.200
5C......	80 000	88 000	80 000	88 000	80 000	0.200	0.200	0.200	0.200

TEST RESULTS

Test	Number of Specimens	$\Sigma n/N$ from Test				Predicted $\Sigma n/N$		
		Min	Max	Avg	Median	Miner	Henry	ASTM Paper
3A.........	4	1.346	1.854	1.644	1.688	1.000	1.218	1.093
3B.........	4	0.769	0.818	0.787	0.781	1.000	0.785	0.905
5A.........	5	0.893	1.353	1.107	1.032	1.000	1.207	1.091
5B.........	5	0.738[a]	0.996	0.875	0.907	1.000	0.841	0.915
5C.........	4	0.728[b]	0.917	0.846	0.869	1.000	0.928	0.981

[a] Two out of five specimens failed in fourth step.

[b] One out of four specimens failed in fourth step.

fact, the recognition of certain limitations in the latter's method that led to this research program. In order adequately to consider Mr. Benedicto's comments, two general considerations must be discussed. The first deals with the basic implications of the Henry method. The second deals with the suitability of the specific data, that of TN 4284, chosen by Mr. Benedicto for comparing the methods in question.

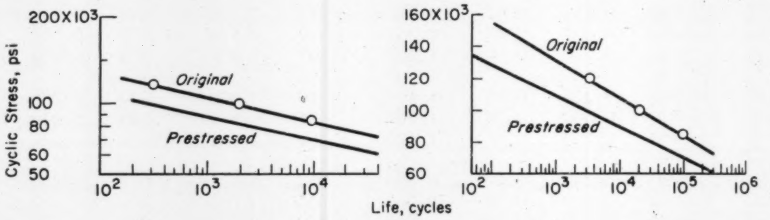

FIG. 10.—Effect of Constant Stress-Concentration Factor on Prestress S-N Line Using a Log-Log and a Semilog-type Plot. (Data from this investigation.)

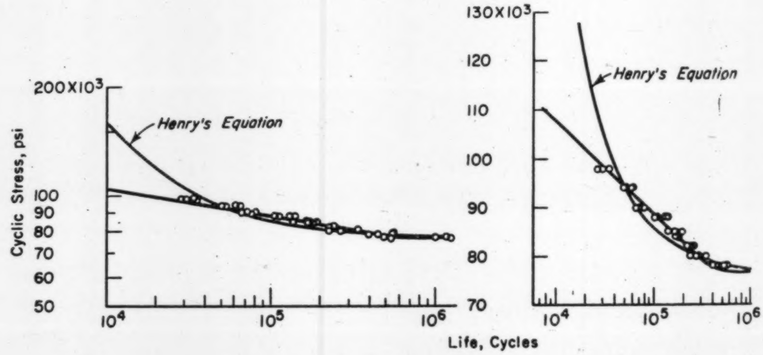

FIG. 11.—Original Material Data from NACA TN 4284 Showing Cyclic Stress and Cyclic Life Relation Expressed by Henry's Equation and by Straight Line.

Consider first the basic concepts underlying Henry's method. Henry suggests that an effective stress concentration factor is introduced by initially prestressing a material. This factor is considered to be constant over the entire applied stress range. It may be defined as the ratio of the prestress level to the stress level which must be applied to the original material in order to obtain the same life as that which remains after prestressing. The effect of this assumption upon the cyclic stress, cyclic life relationship of prestressed material relative to that of the original material is illustrated in the accompanying Fig. 10. The original material data and the 100,000 psi, 1.7 × 10^4 cycle prestress data obtained in the present investigation are used. When these data are plotted on a semilog scale and the original material cyclic stress - cyclic life relation is expressed by a straight line, that of the prestressed material is a straight line also, which diverges from the original material line at the higher stresses. On a log-log type of plot, parallel lines result. The former case is exactly contrary to the data obtained both by the authors and by Bennett (reference (10) of the paper), who show that a convergence takes place at the higher stresses. The latter case, parallelism of log S - log N lines, is similar to the results obtainable by Miner's hypothesis and is closer to what experimental data show, yet still not ultimately correct.

Another assumption introduced by Henry which must also be considered is that a hyperbolic form of equation describes the fatigue behavior of the original material. This evidently is not the case for many materials. That it is not so for 4130 steel, fatigue tested in rotating bending, is evident from Fig. 4 of this paper. The original material data of TN 4284, also for 4130 steel and referred to by Mr. Benedicto, are plotted here in Fig. 11. Both a semilog and a log-log type scale were used. It is evident that a straight line and not a hyperbola best fits the data in each case.

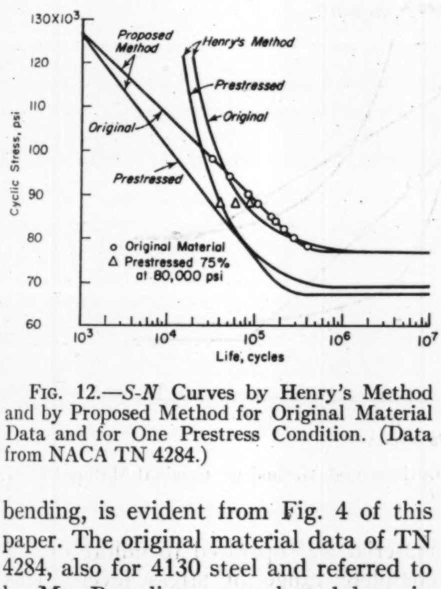

FIG. 12.—S-N Curves by Henry's Method and by Proposed Method for Original Material Data and for One Prestress Condition. (Data from NACA TN 4284.)

The method of Henry combines these assumptions in deriving the equations for damage incurred by prestressing. These equations were applied to predict the cyclic stress - cyclic life relationship for the prestress conditions of TN 4284 cited by Mr. Benedicto and the results are shown in the next two figures.

Figure 12 illustrates the 75 per cent prestress condition at 80,000 psi followed by operation to failure at 88,000 psi. It is again evident that a straight line and not a hyperbola best describe the original material data. Also, the S - log N curve for the prestressed material, as predicted by Henry's method, converges with and eventually crosses the straight line which best fits the original material data. That the linearity of the original material data persists to much higher stress levels with this material is indicated by our data (Fig. 4 of this paper) up to 120,000 psi. Thus, at higher stress levels, yet well below the ultimate strength of the material, Henry's method predicts a value of fatigue life as long, or longer, than that expected for the original material. Figure 13 indicates that a similar situation occurs for the other prestress condition cited by Mr. Benedicto from the data of TN 4284. The S - log N line for the prestressed material as predicted by Miner's method has not been included on these figures for the sake of clarity. It would lie parallel to that of the original material and pass through the point of prestress. From the foregoing com-

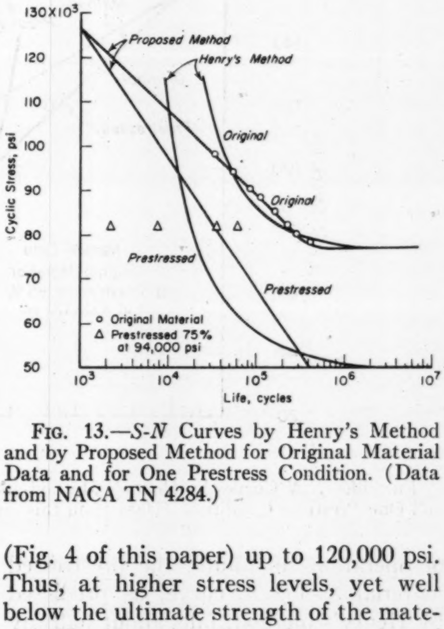

FIG. 13.—S-N Curves by Henry's Method and by Proposed Method for Original Material Data and for One Prestress Condition. (Data from NACA TN 4284.)

parisons, it may be concluded that the method of Henry has a limited range of applicability, and is most useful in the region near the knee of the S - log N curve.

The second general consideration which should be discussed is the suitability of the specific data chosen by Mr. Benedicto in comparing the various methods of predicting fatigue life. It will be noted from Fig. 13 that at the point of operation to failure, the prestressed material S - log N curve, as predicted by Henry's method, falls about halfway between the 4 data points plotted. That of the proposed method falls a little to the right of Henry's curve. In this case, as well as that shown in Fig. 12, the degree of scatter and especially the limited number of data points involved make it difficult to determine the degree of agreement or lack of it between theory and experiment. It should be noted that the authors found that considerably more than 4 data points were required to adequately determine fatigue behavior at a given stress level after prior stressing. The fact that a comparison can only be made at a single stress level after prestressing further limits the usefulness of this comparison. It would appear then that the data of TN 4284 are not a suitable vehicle for comparing the validity of these methods of predicting fatigue life. A more meaningful comparison may be obtained using the data of this investigation, in which the prestressed material was operated to failure over a complete range of stress levels. This comparison is made in Fig. 14. The 100,000 psi prestress condition for 1.7 × 10^4 cycles was chosen. The generally good agreement of the proposed procedure with experimental data over a complete range of stress levels is markedly evident, as is the limited range of applicability of Henry's method.

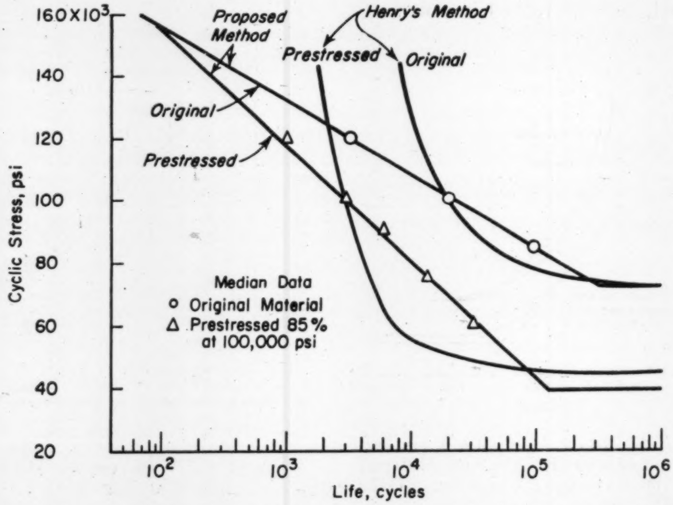

FIG. 14.—S-N Curves by Henry's Method and by Proposed Method for Original Material Data and One Prestress Condition. (Data from this paper.)

In regard to Mr. Benedicto's final comment that neither Henry's nor the proposed method can be used to predict the fatigue life of an actual structure in service when the loads are applied in a random manner, it should be noted that an approximate knowledge of the random load likely to be encountered in actual service will permit calculation of the remaining fatigue life by the proposed method. It will be recalled that Eq 2 of the paper describes the general case for any number of prestress conditions. This equation accounts for the effect of the order-dependency of load application, and any load pattern may be inserted and remaining life calculated. For a complex load history, use of modern computing methods would make such a calculation a relatively easy one.

MR. CLARENCE R. SMITH.[2]—Although good correlation is shown by the authors for smooth specimens, for a full-scale structure, the stress at a concentration suffers plastic deformation on a high load so that subsequent loading is not a function of the load level but some other value which considers the residual stress caused by localized yielding. Also, for a complete structure, say, in a riveted joint structure, reversal of load does not reverse the stress but introduces repeated stresses, not necessarily of the same magnitude. I think the method proposed, if used with true stress at the concentration, shows great promise. The fact that a good correlation was obtained on the smooth specimens does not mean that we could predict the life of a full-scale structure without a lot more knowledge as to the actual stress occurring at points of concentration.

MR. FRECHE.—The authors recognize that the experimental conditions of the present investigation are not necessarily indicative of actual structural applications. This is particularly true with regard to the loading spectra applied as well as the unnotched type of specimens employed. However, the simplified type of test conducted in the present investigation certainly sets forth the minimum requirements that the proposed method must satisfy.

Further investigation is obviously needed to establish the usefulness of the proposed method, particularly with regard to its application to full-scale structures. For example, it is necessary to determine the significance of the constant, N_P, in order to establish whether it is related primarily to the type of material or the type of fatigue loading. Equally important is the need to conduct fatigue studies with other types of materials. Also, it is necessary to investigate other conditions of fatigue loading and loading spectra such as might be encountered in a full-scale structure with attendant stress concentrations. Only then can the applicability of the proposed method to a complex type of structure such as that mentioned by Mr. Smith be established.

MR. C. E. WORK.[3]—The idea of expressing cumulative damage in fatigue by a new relation which is simple enough to be of practical use and yet free from some of the basic weaknesses of the commonly used Palmgren-Miner procedure is worthy of serious consideration. Manson and his associates have presented a very interesting and convincing case for the method they propose. In particular, the idea "that the S - log N line for a prestressed material would tend to converge toward the S - log N line of the original material in the high stress region" is one that seems well founded in fact.

The location of that point of convergence may not, however, be as simple as assumed by Manson and his colleagues. Evidence obtained in the laboratories of the Michigan College of Mining

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[2] Design Specialist, Convair, Division of General Dynamics, San Diego. Calif.

[3] Professor and Head, Department of Engineering Mechanics, Michigan College of Mining and Technology, Houghton, Mich.

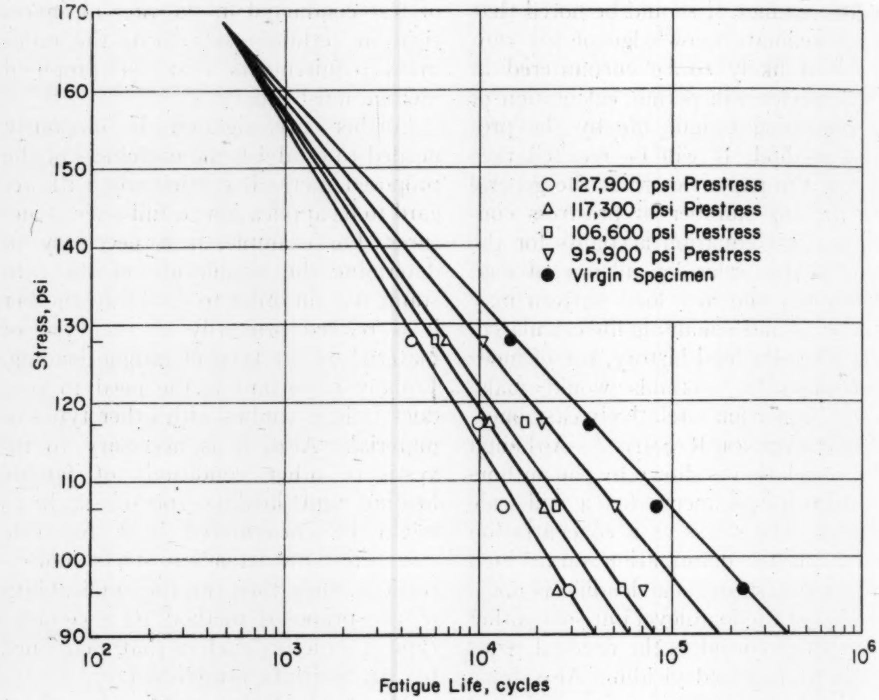

FIG. 15.—Convergence of S - log N Lines for 70 per Cent Cycle Ratio Prestress.

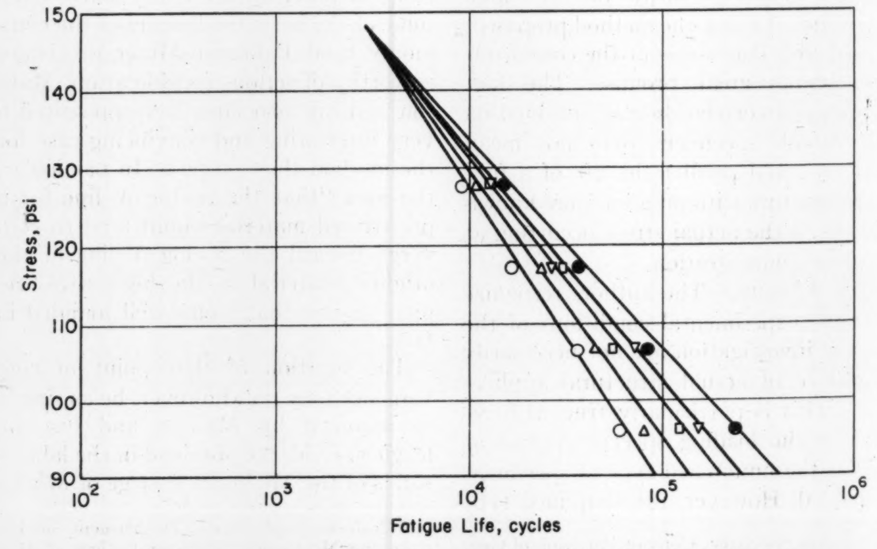

FIG. 16.—Convergence of S - log N Lines for 35 per Cent Cycle Ratio Prestress.

and Technology[4] provides an additional opportunity to test this assumption. The experiments were on SAE 4340 steel specimens subjected to rotating bending in a Krouse high-speed cantilever fatigue testing machine. The steel was heat treated to a tensile strength of 156,000 psi with a yield point of 145,000 psi. The procedures used were described in a paper presented to the Society in June, 1961.[5]

Figure 15 shows the original *S-N* line and damage lines for four sets of specimens prestressed to 70 per cent cycle ratio at stresses 12.5, 25, 37.5, and 50 per cent above the virgin endurance limit respectively. Each point plotted represents the antilog of the mean logarithm of life of fifteen specimens. Estimation of the best intersection of the lines, if done by eye, is subject to considerable personal judgment. For the 70 per cent cycle ratio prestress the writer selected a point P_{70} at $\sigma = 169{,}000$, $N = 4.8 \times 10^2$. This point was chosen as halfway between what he considers the extremes of possible interpretation; that is, it seems unlikely that individual judgment in interpreting these data could possibly differ more widely than P_{70}^{+} at $\sigma = 182{,}000$, $N = 1.8 \times 10^2$, and P_{70}^{-} at $\sigma = 156{,}000$, $N = 1.3 \times 10^3$.

The data for 35 per cent cycle ratio plotted in Fig. 16 appears to intersect at a lower point P_{35} at $\sigma = 148{,}000$ psi, $N = 2.9 \times 10^3$ chosen as midway between P_{35}^{+} at $\sigma = 162{,}000$, $N = 9.6 \times 10^2$ and P_{35}^{-} at $\sigma = 134{,}000$, $N = 8.8 \times 10^3$ which appear to be the most divergent points which could possibly be considered for intersection of the 35 per cent cycle ratio data.

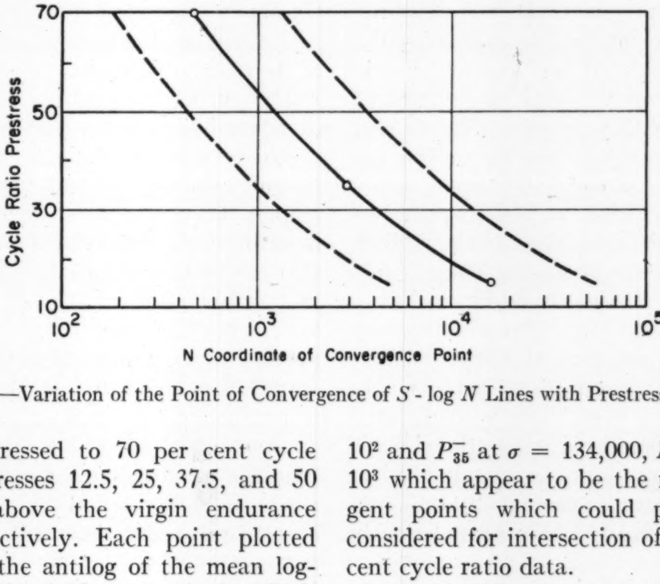

FIG. 17.—Variation of the Point of Convergence of *S* - log *N* Lines with Prestress Cycles.

Data for 15 per cent cycle ratio and 5 per cent approach the virgin *S* - log *N* line and, in fact, overlap and cross it to the extent that the possibility of selecting a single point of convergence is questionable. However, on a graph plotted for 15 per cent cycle ratio prestress, not presented here, an intersection point P_{15} with coordinates $\sigma = 127{,}500$ psi and $N = 1.6 \times 10^4$ was chosen. Overlap was so extensive for the 5 per cent cycle ratio of prestress that it did not seem practical to attempt to define an intersection point for the 5 per cent data.

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[4] W. H. Erickson, "A Quantitative Evaluation of the Damage Produced in SAE 4340 Steel by Repeated Stressing," M.S. Thesis, Department of Engineering Mechanics, Michigan College of Mining and Technology, 60 pp. (1960).

[5] W. M. Erickson and C. E. Work, "A Study of the Accumulation of Fatigue Damage in Steel," see p. 704, this publication.

parisons, it may be concluded that the method of Henry has a limited range of applicability, and is most useful in the region near the knee of the S - log N curve.

The second general consideration which should be discussed is the suitability of the specific data chosen by Mr. Benedicto in comparing the various methods of predicting fatigue life. It will be noted from Fig. 13 that at the point of operation to failure, the prestressed material S - log N curve, as predicted by Henry's method, falls about halfway between the 4 data points plotted. That of the proposed method falls a little to the right of Henry's curve. In this case, as well as that shown in Fig. 12, the degree of scatter and especially the limited number of data points involved make it difficult to determine the degree of agreement or lack of it between theory and experiment. It should be noted that the authors found that considerably more than 4 data points were required to adequately determine fatigue behavior at a given stress level after prior stressing. The fact that a comparison can only be made at a single stress level after prestressing further limits the usefulness of this comparison. It would appear then that the data of TN 4284 are not a suitable vehicle for comparing the validity of these methods of predicting fatigue life. A more meaningful comparison may be obtained using the data of this investigation, in which the prestressed material was operated to failure over a complete range of stress levels. This comparison is made in Fig. 14. The 100,000 psi prestress condition for 1.7 × 10^4 cycles was chosen. The generally good agreement of the proposed procedure with experimental data over a complete range of stress levels is markedly evident, as is the limited range of applicability of Henry's method.

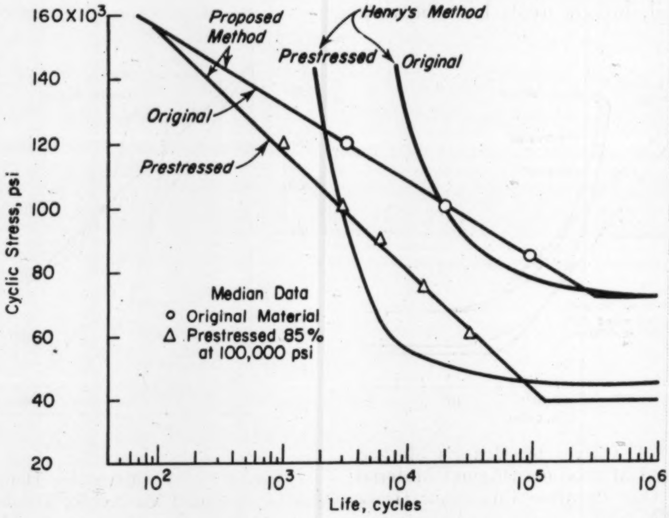

FIG. 14.—S-N Curves by Henry's Method and by Proposed Method for Original Material Data and One Prestress Condition. (Data from this paper.)

In regard to Mr. Benedicto's final comment that neither Henry's nor the proposed method can be used to predict the fatigue life of an actual structure in service when the loads are applied in a random manner, it should be noted that an approximate knowledge of the random load likely to be encountered in actual service will permit calculation of the remaining fatigue life by the proposed method. It will be recalled that Eq 2 of the paper describes the general case for any number of prestress conditions. This equation accounts for the effect of the order-dependency of load application, and any load pattern may be inserted and remaining life calculated. For a complex load history, use of modern computing methods would make such a calculation a relatively easy one.

MR. CLARENCE R. SMITH.[2]—Although good correlation is shown by the authors for smooth specimens, for a full-scale structure, the stress at a concentration suffers plastic deformation on a high load so that subsequent loading is not a function of the load level but some other value which considers the residual stress caused by localized yielding. Also, for a complete structure, say, in a riveted joint structure, reversal of load does not reverse the stress but introduces repeated stresses, not necessarily of the same magnitude. I think the method proposed, if used with true stress at the concentration, shows great promise. The fact that a good correlation was obtained on the smooth specimens does not mean that we could predict the life of a full-scale structure without a lot more knowledge as to the actual stress occurring at points of concentration.

MR. FRECHE.—The authors recognize that the experimental conditions of the present investigation are not necessarily indicative of actual structural applications. This is particularly true with regard to the loading spectra applied as well as the unnotched type of specimens employed. However, the simplified type of test conducted in the present investigation certainly sets forth the minimum requirements that the proposed method must satisfy.

Further investigation is obviously needed to establish the usefulness of the proposed method, particularly with regard to its application to full-scale structures. For example, it is necessary to determine the significance of the constant, N_P, in order to establish whether it is related primarily to the type of material or the type of fatigue loading. Equally important is the need to conduct fatigue studies with other types of materials. Also, it is necessary to investigate other conditions of fatigue loading and loading spectra such as might be encountered in a full-scale structure with attendant stress concentrations. Only then can the applicability of the proposed method to a complex type of structure such as that mentioned by Mr. Smith be established.

MR. C. E. WORK.[3]—The idea of expressing cumulative damage in fatigue by a new relation which is simple enough to be of practical use and yet free from some of the basic weaknesses of the commonly used Palmgren-Miner procedure is worthy of serious consideration. Manson and his associates have presented a very interesting and convincing case for the method they propose. In particular, the idea "that the S - log N line for a prestressed material would tend to converge toward the S - log N line of the original material in the high stress region" is one that seems well founded in fact.

The location of that point of convergence may not, however, be as simple as assumed by Manson and his colleagues. Evidence obtained in the laboratories of the Michigan College of Mining

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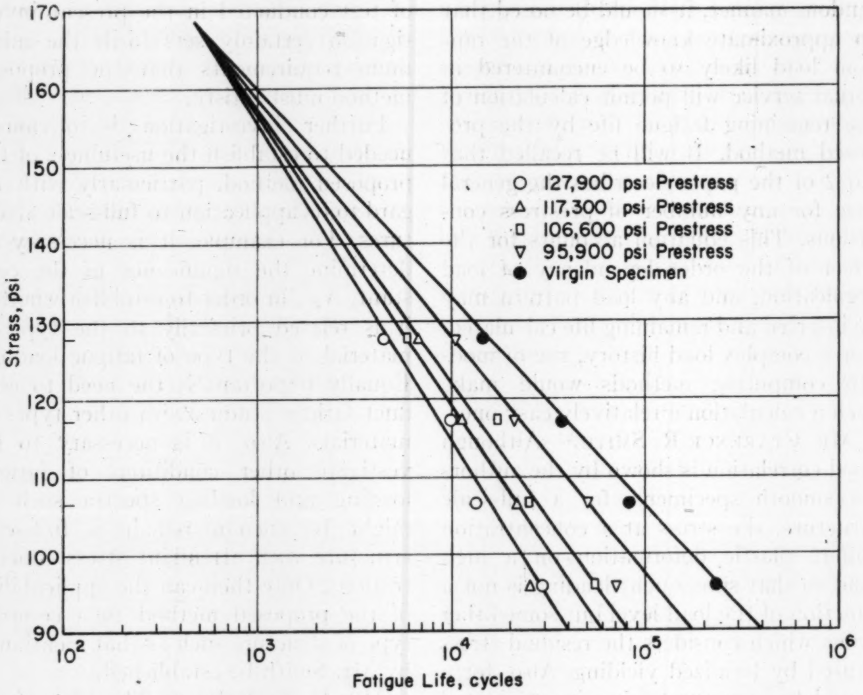

FIG. 15.—Convergence of S - log N Lines for 70 per Cent Cycle Ratio Prestress.

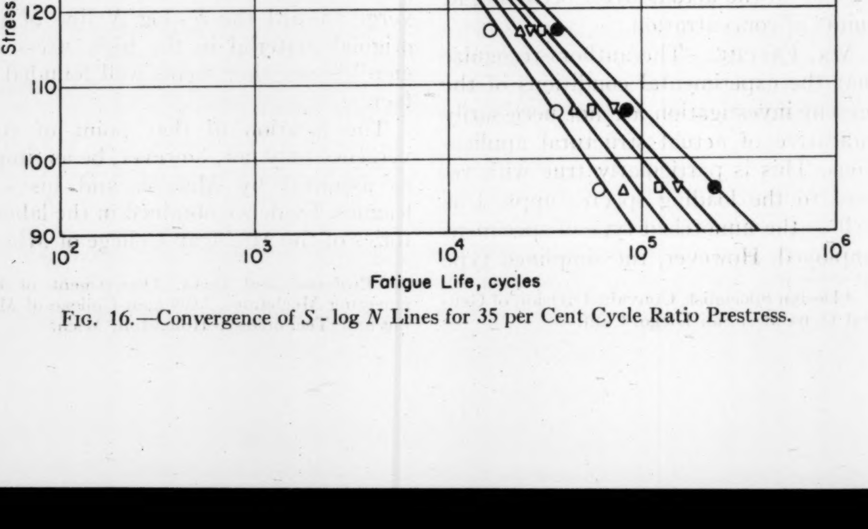

FIG. 16.—Convergence of S - log N Lines for 35 per Cent Cycle Ratio Prestress.

and Technology[4] provides an additional opportunity to test this assumption. The experiments were on SAE 4340 steel specimens subjected to rotating bending in a Krouse high-speed cantilever fatigue testing machine. The steel was heat treated to a tensile strength of 156,000 psi with a yield point of 145,000 psi. The procedures used were described in a paper presented to the Society in June, 1961.[5]

Figure 15 shows the original *S-N* line and damage lines for four sets of specimens prestressed to 70 per cent cycle ratio at stresses 12.5, 25, 37.5, and 50 per cent above the virgin endurance limit respectively. Each point plotted represents the antilog of the mean logarithm of life of fifteen specimens. Estimation of the best intersection of the lines, if done by eye, is subject to considerable personal judgment. For the 70 per cent cycle ratio prestress the writer selected a point P_{70} at $\sigma = 169{,}000$, $N = 4.8 \times 10^2$. This point was chosen as halfway between what he considers the extremes of possible interpretation; that is, it seems unlikely that individual judgment in interpreting these data could possibly differ more widely than P_{70}^{+} at $\sigma = 182{,}000$, $N = 1.8 \times 10^2$, and P_{70}^{-} at $\sigma = 156{,}000$, $N = 1.3 \times 10^3$.

The data for 35 per cent cycle ratio plotted in Fig. 16 appears to intersect at a lower point P_{35} at $\sigma = 148{,}000$ psi, $N = 2.9 \times 10^3$ chosen as midway between P_{35}^{+} at $\sigma = 162{,}000$, $N = 9.6 \times 10^2$ and P_{35}^{-} at $\sigma = 134{,}000$, $N = 8.8 \times 10^3$ which appear to be the most divergent points which could possibly be considered for intersection of the 35 per cent cycle ratio data.

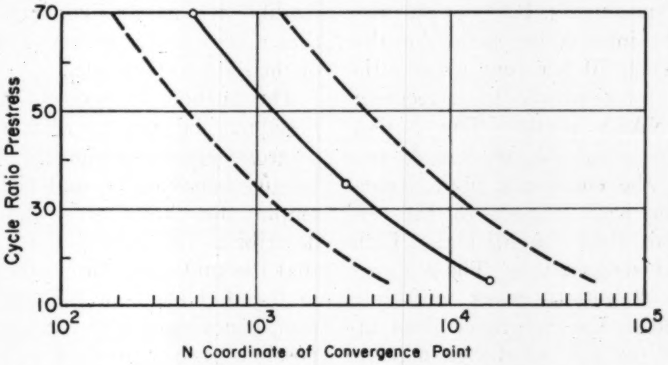

FIG. 17.—Variation of the Point of Convergence of *S* - log *N* Lines with Prestress Cycles.

Data for 15 per cent cycle ratio and 5 per cent approach the virgin *S* - log *N* line and, in fact, overlap and cross it to the extent that the possibility of selecting a single point of convergence is questionable. However, on a graph plotted for 15 per cent cycle ratio prestress, not presented here, an intersection point P_{15} with coordinates $\sigma = 127{,}500$ psi and $N = 1.6 \times 10^4$ was chosen. Overlap was so extensive for the 5 per cent cycle ratio of prestress that it did not seem practical to attempt to define an intersection point for the 5 per cent data.

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[4] W. H. Erickson, "A Quantitative Evaluation of the Damage Produced in SAE 4340 Steel by Repeated Stressing," M.S. Thesis, Department of Engineering Mechanics, Michigan College of Mining and Technology, 60 pp. (1960).

[5] W. M. Erickson and C. E. Work, "A Study of the Accumulation of Fatigue Damage in Steel," see p. 704, this publication.

A general trend, however, seems to be apparent and consists of a shifting of the point of convergence toward lower stresses and longer life as the cycle ratio of prestress is increased. This trend is illustrated in Fig. 17 by the heavy median curve shown. Two other lighter lines outline a scatter band representing the widest differences of opinion imaginable in interpreting points of convergence of the S - log N lines.

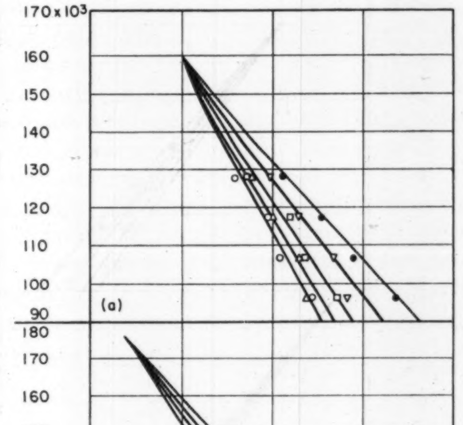
The NASA tests utilized cycle ratios of 50, 70, and 85 per cent which were somewhat higher than those in the Michigan Tech experiments. It is not surprising that the intersection point for the Michigan Tech 70 per cent cycle ratio tests were most nearly in agreement with the NASA results. The NASA stresses were about 17, 38, and 65 per cent above the endurance limit, somewhat higher with respect to the endurance limit than the Michigan Tech spectrum of stress levels. These stress levels were chosen to cover uniformly the range from the endurance limit up to about 90 per cent of the yield point of the steel being used. Appreciably higher stresses would have been expected to exceed the static proportional limit and thus leave stress calculations in bending open to question. Static properties of the NASA steel were apparently not reported.

The influence of prestressing on endurance limit is now being investigated as an extension of the Michigan Tech research. Information about the life at which the knee in the S - log N curve occurs will be available when these experiments are completed. Although endurance limit tests are very time-consuming, even in high-speed machines, it is hoped that this work may be completed in time for presentation before the Society in 1962.

MR. FRECHE.—The authors welcome the added corroboratory evidence for the S - log N line convergence concept provided by Professor Work's data. With regard to Professor Work's comment as to the actual location of the point of convergence, it would appear from his data that it does indeed shift toward lower stresses and longer life as the cycle ratio of prestress is increased. The authors do not have sufficient data either to verify or contradict Mr. Work's conclusion on this point. However, the significant question at issue is not the exact location of the point of convergence. It is rather whether or not it is possible to select readily a common convergence point that allows a satisfactory representation of the data to be made.

The authors recognize that the proposed method suggests what is essentially a gross approximation of the actual fatigue behavior of materials. For example, the choice of straight lines for describing the behavior of a material that has undergone prior stressing, made in the derivation of the proposed relation, is not rigorously correct. Since it is possible to so approximate the S - log N curves of many materials without sacrificing significant accuracy, the approximation was made to simplify the derivation of the equations. It should be noted that such a derivation could also have been made and the same relation would have resulted if linearity would have occurred on any coordinate system of stress versus log N.

The choice of the common convergence point of the S - log N lines should be viewed in the same vein. It will be recalled that the authors suggest the commonly used approximation of their reference (11) to arrive at a reasonable value of N_P for steels when experimental facilities are lacking. This reference indicates convergence of the log S - log N lines for variously surface treated (surface damaged) steels at approximately 1000 cycles. The data of the present investigation, when plotted on a log-log scale, similarly result in convergence at 1000 cycles. The latter value would thus appear to be a reasonable choice when experimental facilities are lacking. Finally, it should be noted that calculations by the proposed relation of remaining fatigue life after prior stressing in rotating bending with SAE 4130 steel indicate that the choice of the point of convergence has no marked effect upon predicted life when N_P is chosen as 1000 cycles, or as low as 100 cycles. Thus, the user of the proposed relation should



(*a*) Common intersection point; 1000 cycles.

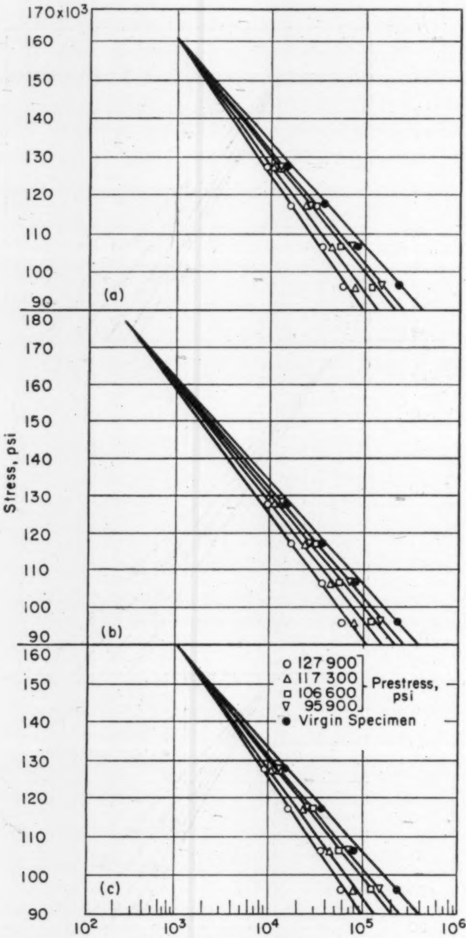
(*b*) Common intersection point; intersection of original material S - log N line and line drawn through data representing remaining life at the highest and lowest stress considered after prestressing 70 per cent at 127,900 psi.

(*c*) Common intersection point; average of the convergence points selected by Professor Work for the 35 per cent and the 70 per cent prestress conditions.

FIG. 18.—Convergence of S - log N Lines for 70 per Cent Cycle Ratio Prestress.

have a rather wide latitude in the matter of selecting the convergence point, N_P.

Although Professor Work's data indicate an apparent shifting of the point of convergence toward lower stresses and longer life as the prestress cycle ratio is increased, it can be shown that these data can be described quite well by lines which pass through different common convergence points obtained by various approximations. Only the 70 and the 35 per cent cycle ratio prestress data are used to illustrate this fact, since both the 15 and 5 per cent data (according to Professor Work) overlapped the virgin S - log N line to such an extent that selection of a convergence point was questionable in these instances.



(*a*) Common intersection point; 1000 cycles.
(*b*) Common intersection point; same as on Fig. 18(*b*).
(*c*) Common intersection point; same as on Fig. 18(*c*).

FIG. 19.—Convergence of S - log N Lines for 35 per Cent Cycle Ratio Prestress.

When 1000 cycles are arbitrarily selected as the common convergence point, it is evident from the accompanying Figs. 18(*a*) and 19(*a*) that the *S* - log *N* lines can be drawn so as to pass through this particular point and the Michigan College of Mining and Technology (MCMT) data with approximately the same degree of certitude as those drawn by Professor Work.

By using still another method of approximation, a similar result will be observed. This approximation method consists of drawing a line through the data representing remaining life at the highest and lowest stresses considered after prestressing 70 per cent at 127,900 psi, and noting its intersection with the original material *S* - log *N* line. By so doing, the largest possible displacement is obtained between the prestress and the original material *S* - log *N* lines, thus tending to provide a maximum degree of confidence in the intersection point. This graphical approximation of N_P is analogous to the manner in which the common convergence point might be obtained when only limited experimental tests can be made. Figures 18(*b*) and 19(*b*) indicate that the *S* - log *N* lines drawn through the MCMT data and emanating from a common convergence point so obtained also fit the data well.

A final interesting approximation may be made by taking an average of the coordinates of the convergence points specified by Professor Work for the 70 and the 35 per cent prestress conditions. The resulting coordinates of the new common convergence point are $\sigma =$ 158,500 psi, and $N = 1177$ cycles (average of the log cyclic lives). Figures 18(*c*) and 19(*c*) provide plots of the MCMT data as well as the common convergence point obtained in this manner. Again, it is evident that the *S* - log *N* lines can be drawn to fit the data well and still meet at this point of convergence.

From the preceding it would appear reasonable to conclude that the premise of the proposed method, namely the use of a practical compromise in selecting a common convergence point of *S* - log *N* lines, is feasible with SAE 4130 steel in rotating bending fatigue. The data provided by Professor Work tend to substantiate the assumptions made by the authors in this regard. Finally, the need for an exact determination of the point of convergence as implied by Professor Work, which generally involves an extensive experimental program, would seem to be warranted only to establish the validity of the convergence concept in instances where a completely different type of material or a different type of fatigue stressing is involved.

A STUDY OF THE ACCUMULATION OF FATIGUE DAMAGE IN STEEL*

By William H. Erickson[1] and Clyde E. Work[2]

Synopsis

A study was made of the accumulation of fatigue damage in SAE 4340 steel at stresses up to 150 per cent of the endurance limit. It was observed that damage was not unique but depended on the stresses employed to evaluate it, and it was not a linear function of the number of repetitions of stress.

The observed behavior was explained in terms of a characteristic number of crack nuclei formed by initial cycles of stress and propagation of cracks from these nuclei. The number of crack nuclei formed was controlled by the initial stress amplitude. The rate of crack propagation was controlled by the stress amplitude during later cycles. Total life was governed by both factors.

The prediction of the fatigue life of structural components subjected to repeated stresses of varying amplitude is a major unresolved design problem. The ordinary *S-N* curve for constant stress amplitudes is the most common form in which fatigue data are available. However, it is not clear how this simple kind of information can be used to predict the behavior of members subjected to stress histories of mixed amplitude. Use of the *S-N* curve obtained for virgin specimens is complicated by the fact that applying even a few cycles of prestress to a set of specimens usually alters the whole curve.

The failure of many attempts to predict life on the basis of the *S-N* curve can be attributed to the necessity of assuming some unique rate of accumulation of damage for each given stress level. Unfortunately, the damaging effect of a given stress cycle is a function of both prior and future stress history.

It has been found that after a relatively few cycles of applied stress, localized broadened slip bands appear within the grains followed by cracks which can be observed microscopically to develop along these bands. When and how these cracks begin is not clearly known. However, there is evidence that the broadened slip bands, or at least some of them, actually contain a microcrack after a very few stress cycles (**1**).[3] The slip-band markings which ultimately yield observable cracks cannot be etched away, while surrounding slip bands disappear under the etching action (**2**). This indicates that some significant structural change is already present at that early stage. If the broadened slip band did not contain permanently damaged structure in the form of submicroscopic cracks, it would be expected that a recrystallization heat treatment would restore the material to its original undamaged state. However, this is not the case (3).

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* Presented at the Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

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[2] Professor and Head, Department of Engineering Mechanics, Michigan College of Mining and Technology, Houghton, Mich.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

Forsyth's work (4) leads to the observation that the spacing between these broadened slip bands is determined by the spacing of the slip bands produced by the first few stress cycles and that the number of deformed zones is proportional to the stress level in the region involved.

While it appears that microcracks are produced by the first few stress repetitions, their extension is a slow process which may comprise the largest part of the fatigue life. On reaching some critical length, or probably upon joining other "cracklets," the cracks propagate rather precipitously to complete fracture.

The number of stress repetitions of varying amplitude to failure can probably be predicted best at the present time by empirical or semiempirical expressions developed from observations of large-scale behavior of a particular metal when subjected to selected sequences or stress repetition. However, no completely satisfactory expressions have yet been developed. Experiments have followed three general approaches:

1. Observations of the effect of one or more blocks of constant stress cycling on the life at other stress levels or on the endurance limit have been attempted with the hope that a unique damage value could be found for a given number of repetitions of a particular stress, regardless of when in the total life they are applied. This would have made it possible to compute the number of repetitions to failure for particular combinations of certain stress amplitudes. The well-known Miner rule (5) is based on this assumption that a unique degree of damage is caused by one stress cycle of a given amplitude whenever in the stress history it was applied. In view of the continuous changes occurring in the structure, however, the damaging effect of any one stress cycle of any particular amplitude is not unique, but depends on the state of damage at that instant.

Richart and Newmark (6) suggested a method for estimating the fatigue life by use of experimentally determined nonlinear relationships between damage and number of cycles imposed.

2. The repeated application of specified patterns of stress amplitudes has been carried out (7,8) in attempts to eliminate the effect of the sequence of testing. In general, only qualitative conclusions have been forthcoming from these repeated pattern tests.

3. Because application of repeated stresses in large blocks of constant amplitude appears to be unlike service conditions and limited by the particular sequences of stressing, randomized sequences of stress amplitudes (9) have been employed in efforts to simulate service conditions in laboratory tests. Not all service conditions involve random stressing, and the rational expression of results from such tests in terms of any so-called fundamental properties or mechanisms has not yet been achieved.

TABLE I.—CHEMICAL ANALYSIS OF STEEL USED.

Element	Per cent
Carbon	0.39
Manganese	0.71
Phosphorus	0.009
Sulfur	0.009
Silicon	0.29
Nickel	1.79
Chromium	0.76
Molybdenum	0.124
Iron	remainder

In this investigation, experiments were performed to observe the influence of various sizes of blocks of cycles of each of four different levels of repeated stress on the subsequent fatigue life evaluated at each of the other levels. A sufficiently large number of specimens were prepared to give some statistical validity to the observations. The results were examined in the light of a hypothesis which attributed damage to two separate phenomena: crack nucleation and crack propagation.

MATERIAL AND EQUIPMENT USED

The experiments in this investigation were conducted with a Krouse high-speed repeated-stress machine of the rotating-cantilever-beam type. Round specimens of SAE 4340 steel $\frac{1}{4}$ in. in diameter at the smallest section were stressed at a speed of about 6300 cpm. The steel had the chemical analysis shown in Table I.

The rods were cut into specimen blanks, then heat treated by oil quenching from 1500 F after $\frac{1}{2}$ hr at temperature, followed by tempering at 1150 F for 2 hr.

The mechanical properties which resulted were: yield point, 145,000 psi; tensile strength, 156,000 psi; and Rockwell hardness, C scale, 27 to 30. Final machining and polishing was done after heat treatment.

TESTING PROGRAM

The preliminary program consisted of evaluating the endurance limit and determining the number of cycles to failure at each of four different stress levels. The four stress levels chosen were 50, 37.5, 25, and 12.5 per cent above the endurance limit; for convenience, they are designated *A*, *B*, *C*, and *D* levels, respectively.

In evaluating the accumulation of fatigue damage, each specimen was subjected to a certain number of cycles, which was less than the number expected to cause failure, at one of the stress levels *A*, *B*, *C*, or *D*. After this prestressing, the amplitude of applied stress was changed to one of the other three levels, and the number of cycles required to cause the specimen to fail at this second level, or test stress, was observed. The percentage by which the life at the test stress exhibited by prestressed specimens was less than the life of virgin specimens at the test stress level was called "per cent damage."

To ensure that all specimens would be equally representative, the sequence of specimen measurement was randomized, the specimens were assigned to their testing levels by random selection, and the testing sequence was randomized.

TABLE II.—RESULTS OF CONSTANT-STRESS-LEVEL TESTS,

Stress Level Designation	Stress, psi	Percentage of Endurance Limit	Life, $\hat{N}$, psi
A	127 900	150	14 750
B	117 300	137.5	37 100
C	106 600	125	83 800
D	95 900	112.5	234 500

Determining the Basic S-N Curve:

Using the staircase method described in the Appendix,[4] 20 specimens were tested in determining the endurance limit. On the basis of a few exploratory tests, a stress of 85,000 psi was taken as the starting level. Specimens withstanding 30,000,000 repetitions of stress were considered to be survivors. The endurance limit thus obtained was 85,300 psi.

Fifteen specimens were tested to failure at each of four stress levels with the results shown in Table II. Since the distribution of log N is more amenable to statistical analysis than that of N, the measure of life used for analysis of data was the antilogarithm of the mean value of log N and is designed here as N. Points plotted as circles in Fig. 1 represent these values. The scatter at each stress level involved in these studies is indicated by vertical checks at N values corresponding to values of log N one standard deviation on either side of mean log N.

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[4] See p. 715.

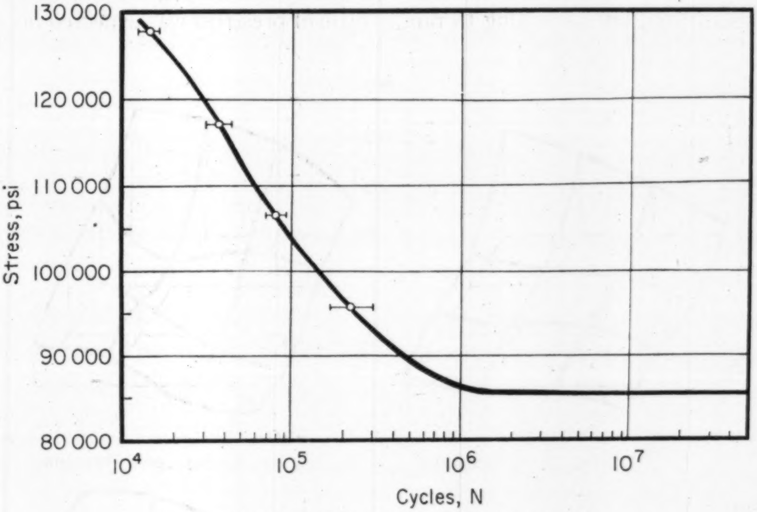

FIG. 1.—Basic *S-N* Curve for SAE 4340 Steel.

Evaluation of Damage from Prestressing:

The damage resulting from prestressing was evaluated in two-level tests. In one series, 30 specimens were prestressed at the D stress level by subjecting them to 5 per cent of $\hat{N}_D$[5] stress repetitions. Immediately following prestressing, the moment on each specimen was changed and the specimen then tested to failure. Ten of the 30 were retested to failure at the A stress level, 10 at the B level, and 10 at the C level. The number of cycles to failure at the test stress was recorded in each case. Similarly, prestress cycles of 15, 35, and 70 per cent of $\hat{N}_D$ were applied to groups of 30 specimens and followed by cycling to failure at the A, B, and C stress levels.

This same test procedure was followed with four groups of 30 specimens each prestressed at stress level C. One group was prestressed 5 per cent, one 15 per cent, one 35 per cent, and one 70 per cent of $\hat{N}_C$ cycles followed by testing 10 from each group to failure at each test stress level A, B, and D. In a similar manner prestresses at B and A levels

TABLE III.—LIFE AFTER PRESTRESSING.

Prestress[a] Test Stress Combination	Remaining Life in Cycles			
	0.05 Cycle Ratio of Prestress	0.15 Cycle Ratio of Prestress	0.35 Cycle Ratio of Prestress	0.70 Cycle Ratio of Prestress
BA.....	15 000	13 000	11 100	6 500
CA.....	14 900	15 800	14 000	5 900
DA.....	15 800	16 100	14 000	10 800
AB.....	33 100	26 100	16 900	10 500
CB.....	35 200	38 200	30 900	17 300
DB.....	39 600	42 000	27 500	21 100
AC.....	76 200	51 640	36 500	13 400
BC.....	77 270	77 450	45 000	22 100
DC.....	89 540	81 300	76 400	50 930
AD.....	135 800	112 200	60 000	28 300
BD.....	161 100	127 300	81 300	25 700
CD.....	190 500	147 900	121 900	55 500

[a] The first letter identifies the prestress, the second the test stress.

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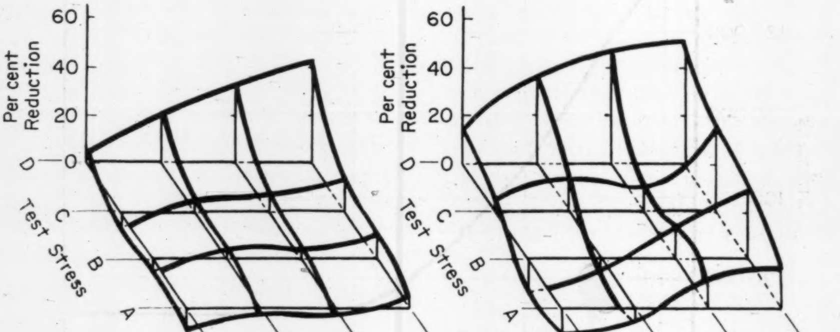
[5] $\hat{N}_D$ represents the mean life to failure at the constant stress amplitude corresponding to the D level.

were applied and followed by test stress levels *A*, *C*, and *D* and *B*, *C*, and *D* respectively.

The actual tests were not run in groups, as might be assumed from the above description, but according to random selection to avoid any systematic error that might arise from the gradual deterioration of the testing machine. In all, 480 specimens were tested in this main area of the investigation. The antilogarithms of the mean of logarithms of cycles to failure at each test stress are given in Table III.



5 per cent Prestress

15 per cent Prestress

35 per cent Prestress

70 per cent Prestress

FIG. 2.—Reduction in Life at Test Stress Resulting from Various Prestress Histories.

It was important to define a representative life value that could be determined even if failure of some specimens were to occur during prestressing. Occasional failure during the 70 per cent cycle ratio of prestress was expected because of normal scatter in fatigue data. A graphical procedure suggested by Roeloffs and Garofalo (10) was followed. This procedure is explained briefly in the Appendix. To check the validity of the graphical procedure, an arithmetic mean of the logarithms was also determined where possible. For the 43 cases where both were available, agreement between the two values was quite good. In 29 cases the difference was 1 per cent or less. In only 3 cases does the difference exceed 4 per cent. To be consistent, the values determined graphically were used for all cases and are the ones listed in Table III.

DISCUSSION OF RESULTS

The influence of prestressing on life at the test stress is illustrated in Fig. 2. In these diagrams the damage is plotted as per cent reduction of life at the various test stresses as functions of prestress amplitude and test stress amplitude. Where the life at the test stress exceeded that of virgin specimens, "negative damage" was plotted, corresponding to an improvement rather than a deterioration of resistance to fatigue stresses.

It will be noted that points on the diagonal connecting the left-rear point to the right-front point, with the base coordinates (D,D), (C,C), (B,B), and (A,A), have equal abscissas defined by the cycle ratio of prestress applied. These correspond to per cent damage equal to the cycle ratio of prestress applied. It is observed that for points behind this diagonal the per cent damage is higher than the cycle ratio applied in prestressing, and in front of that diagonal the per cent damage is less than the cycle ratio applied in prestressing. In some instances, for 5 and 15 per cent cycle ratio prestressing, negative damage results.

The *AA* to *DD* diagonal is the only portion of each graph where a linear accumulation of damage is observed, and this is required by definition of cycle ratio.

Test stresses that are higher than the prestress consistently indicate less damage than predicted by the uniqueness assumption, and lower test stresses indicate greater damage. The results clearly indicate that damage is not a unique quantity, but the damaging effect of a prestress depends on following stress levels.

Two-Stage Crack Hypothesis:

The influence of test stress on the damage observed for a given prestressing, and particularly the instances where prestressing resulted in "negative damage," cannot be explained by any simple damage theory. An explanation of negative damage might be attempted on the basis of strain aging or of relaxation of residual stresses. However, we would expect these phenomena to have a beneficial effect at all subsequent stress levels, not just higher ones.

It appears that the behavior observed is qualitatively consistent with the following explanation in terms of a period of crack nucleation followed by crack growth to failure.

As pointed out earlier, microscopic evidence indicates that the number of crack nuclei formed by the first few cycles depends on the stress level. If this prestress is followed by cycling to failure at a higher stress level, repetitions of the higher stress tend to propagate the damage process primarily at sites already initiated during the first cycles at the lower stress level and not to create the number of sites that would have been initiated in the virgin material by this higher stress. Stress concentration caused by slip at the surface (11,12) could be responsible for localizing further damage at any nominal stress level to nuclei formed early. This possibility is supported by studies of Mott (13), who shows that the stress necessary to move dislocations in a work-hardened lattice is less than that necessary to create new ones. Wood and Davies (14) have shown evidence by X-ray diffraction that alternating slip movements are confined to zones first formed. This restriction of damage has been associated with the formation of intrusions, or crevices (**13,15,16**), which would have a stress-concentrating effect or perhaps merely continue to deepen.

Corten and Dolan (**17**) have suggested an expression $D = mrN^a$ to describe the progress of damage during repeated stressing. Here, D is the damage, N is the number of cycles, r is the coefficient of damage propagation (a function of the stress level), a expresses quantitatively the assumption that the damage rate at a given stress level increases with number of cycles, and m is the number of damage nuclei. This expression assumes that the damage accumulated by a given number of cycles is proportional to the number of crack nuclei extended. The argument could then be made that a few cycles of a low prestress before a higher test stress would cause an increase in cycles to failure at the test stress because of the fewer active damage sites initiated at the lower stress level.

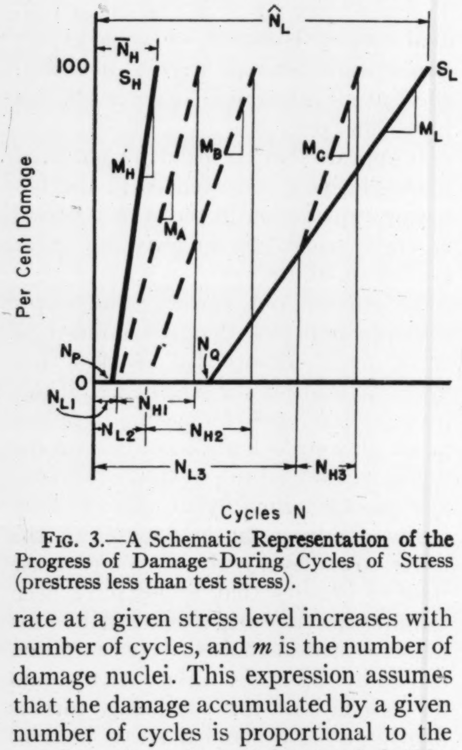


FIG. 3.—A Schematic **Representation of the** Progress of Damage During Cycles of Stress (prestress less than test stress).

The same approach could be used to describe the effect of following the prestress by lower amplitude cycling.

Schematic Representation of Damage:

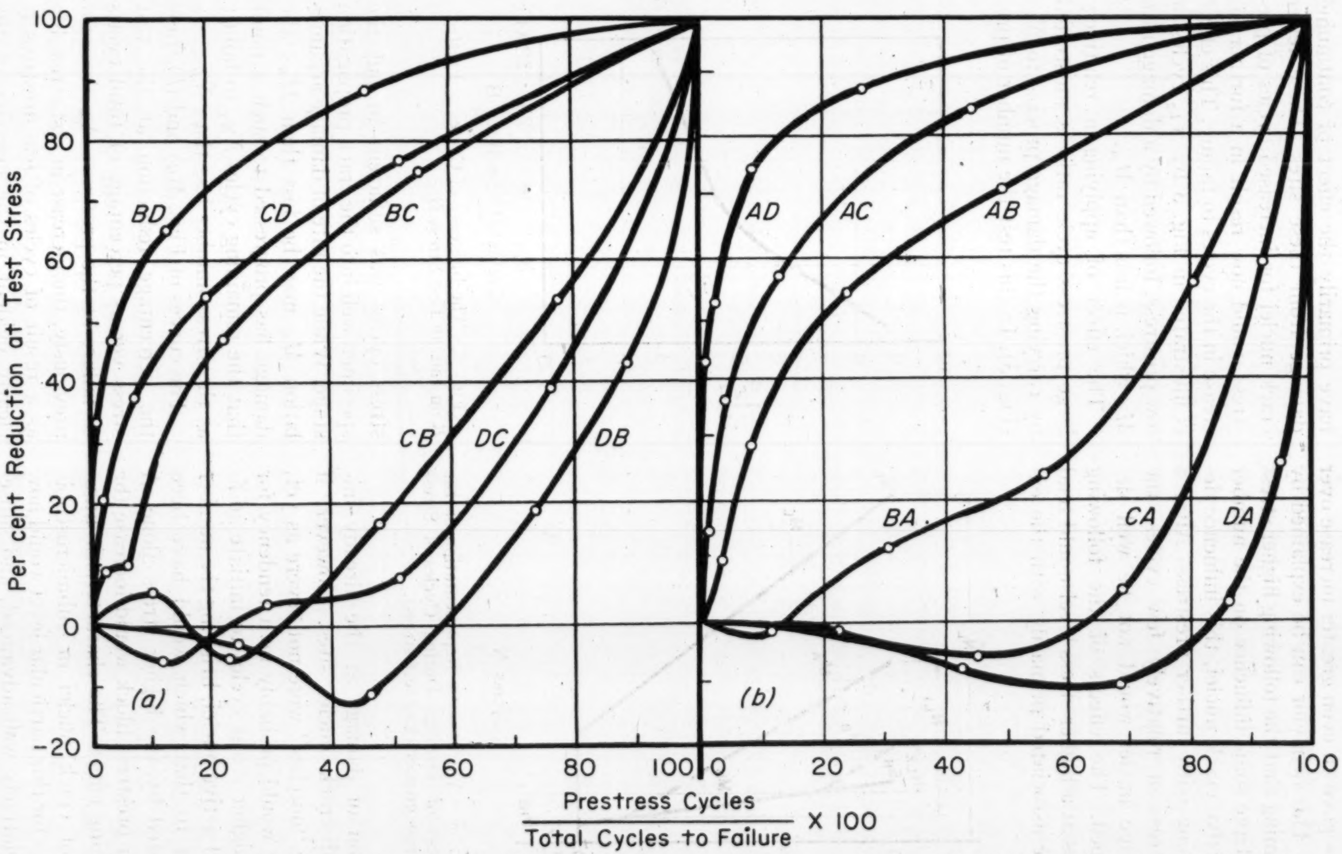
In general, only after some number of initial stress cycles do the subsequent cycles to failure at the higher stress level begin to decrease below $\hat{N}$. What occurs to cause this decrease after both the 5 and 15 per cent prestress histories caused increases?

Figure 3 is a schematic representation of the progress of damage as a function of repeated stress cycles, N, for two stress levels, one high (S_H) and one low (S_L). The shape of the damage curve is based on evidence indicating that the damage process consists of two stages (**18**). The first stage, corresponding to the portion of the curve from the origin to N_p for stress level S_H, and to the portion from the origin to N_Q for S_L, represents crack initiation. The second stage, corresponding to the damage lines of slopes M_H and M_L for stress levels S_H and S_L respectively, represents crack propagation. The linear rates of damage accumulation shown on this sketch perhaps represent an oversimplification. However, these curves would be thought of as average slopes of the actual damage curves.

According to Fig. 3, N_{L_1}, cycles of prestress at the lower stress level S_L, followed by cycling at S_H would result in a damage rate M_A at S_H. This damage rate would be lower than normally found at this stress because of the fewer crack nuclei formed at the lower level. The resulting number of cycles to failure, N_{H_1}, at the higher stress level could conceivably be greater than the $\hat{N}_H$ cycles observed in constant-amplitude tests.

Effect of Further Prestressing:

The cycles to failure at S_H were observed in Fig. 2 to increase still further as a result of a longer prestress at the lower level. Although a 5 per cent prestress generally resulted in an increased life at the test stress, a 15 per cent pre-



(*a*) For stress level combinations *B* and *C*, *C* and *D*, *D* and *B*. (*b*) For stress level combinations *A* and *B*, *A* and *C*, *A* and *D*.

FIG. 4.—The Influence of Various Amounts of Prestress on Change in Remaining Life at Test Stress.

stress gave an even greater increase over $\hat{N}_B$. This behavior can be explained by assuming that the following higher stress can have some influence on the number of active crack nuclei, this influence decreasing with further prestress. After a prestress of relatively few cycles, the damage nuclei would not be well developed. The effects of the following cycles at a higher stress level would tend to be associated primarily with the extension of damage at the already initiated crack nuclei sites. However, if these "notches" were not severe as yet, there would probably be a tendency for the higher stress cycles to initiate additional active nuclei, bringing the number closer to that which would have been initiated by the higher stress alone. A larger prestress block would increase the notching effect. The characteristic number of crack nuclei or submicroscopic cracks for this particular level would now be relatively well advanced, and subsequent cycles at a higher stress would have primarily the effect of continuing damage from these sites. These fewer crack nuclei for increased cycles of prestress would now result in a further increase in the cycles to failure. This effect is illustrated in Fig. 3 by N_{L_2} cycles of low prestress, followed by a damage rate M_B which is less than M_A.

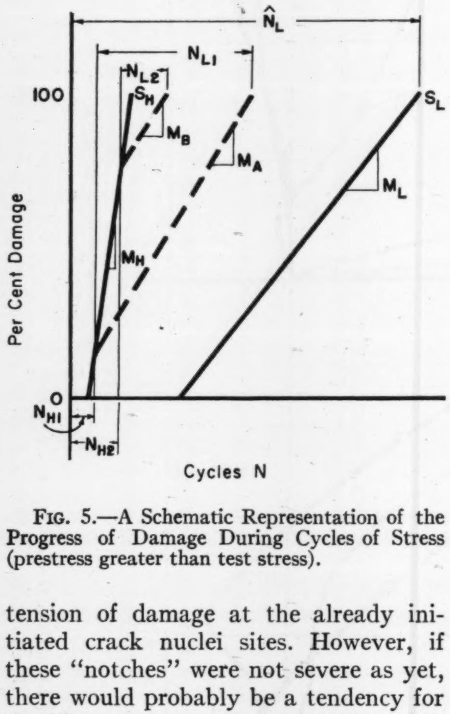


FIG. 5.—A Schematic Representation of the Progress of Damage During Cycles of Stress (prestress greater than test stress).

The effect of applying a relatively large prestress cycle ratio is illustrated by carrying the damage process to N_{L_3} (Fig. 3). In this test the number of prestress cycles was sufficient to put the specimen well into the crack propagation stage. While the rate of damage accumulation M_C may be less than M_B, the damage has progressed to such a point that the remaining cycles N_{H_3} to failure at the stress number less than $\hat{N}_H$.

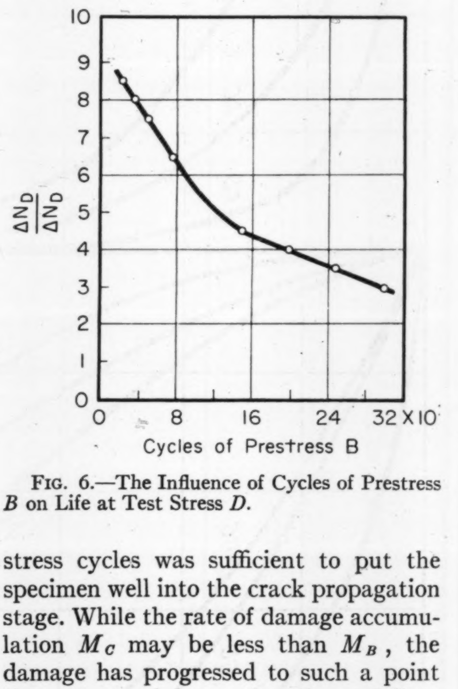


FIG. 6.—The Influence of Cycles of Prestress *B* on Life at Test Stress *D*.

The curves of Figs. 4(*a*) and (*b*) show the percentage reduction at the test stress versus percentage of total cycles to failure at the prestress. As discussed previously, the increase in test stress life as a result of cycles of *low* prestress is clearly evident in the negative of the ordinate.

Prestress Higher than Test Stress:

The curves for prestresses higher than test stresses are characterized by the very high percentage reduction for relatively few prestress cycles. Additional cycles of prestress result in progressively smaller increments of reduction in life at the test stress. The schematic cumulative damage curves in Fig. 5 can be utilized to explain the behavior observed in tests for which the higher stresses were applied first and followed by cycles of lower stress.

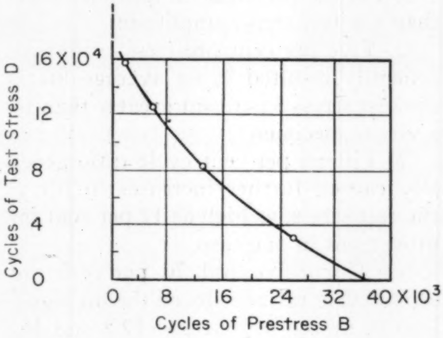
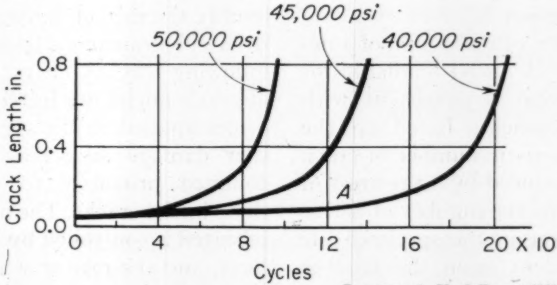


FIG. 7.—The Variation in Rate of Change of Life at Test Stress D with the Number of Cycles of Prestress B.

It can be seen that the extensive reduction in life at S_L after N_{H_1} cycles of prestress S_H does not necessarily indicate that extensive damage has occurred to the material during the prestress. Instead we can assume that the high stress has activated many more crack nuclei or notches than would have occurred if all stresses were at the S_L level. Damage would then accumulate at the more rapid rate, M_A, rather than M_L, with the result of a rather high reduction in life at the test stress.

Following up this argument, Fig. 5 would lead to the expectation that the decrease in the number of test stress cycles should proceed at a constant rate with increased prestressing. If an exponential form of damage curve had been assumed, an increasing rate would be expected. However, the experimental results of this investigation show that with increasing numbers of prestress cycles the percentage reduction in cycles to failure at the test stress goes up at a decreasing rate. This is shown in Fig. 6 as a smooth curve of decreasing negative slope for cycles to failure at the test stress D plotted versus number of cycles of prestress B. The rate of decrease in slope of this curve with additional cycles of prestress is shown in Fig. 7.



Courtesy A. V. deForest (19).

FIG. 8.—The Growth of Cracks with Repetitions of Stress.

This reduction in the rate of decrease of cycles to failure at the lower amplitude test stress would be expected if the schematic damage curves of Fig. 5 could be pictured as having decreasing slopes or if the slope M_B were less than the slope M_A. However, these trends are not consistent with results of investigations on the rates of crack propagation which show a rapidly increasing rate of crack extension with cycling. One graph from deForest (19) is reproduced as Fig. 8 here.

It seems likely, however, that even though the size of any given crack which matures may grow at an increasing rate, before damage reaches the point of selecting one site for completion of fracture the over-all damage rate could show a net decrease if a decreasing number of cracks continued to propagate. This could result from longer cracks relieving the elastic strain from more and more shorter cracks thus stopping their development.

It is assumed here that the longer "cracklets" are still in the slow development stage (crack initiation) as indicated by the letter A in Fig. 8. The effect of the decreasing number of actively developing cracks could be to decrease the rate of damage accumulation.

The introduction of the notion of a decreasing number of nuclei available for continued extension is consistent with the previous arguments based on the idea of a characteristic number of crack nuclei being introduced by a prestress of given amplitude as the number of active nuclei at any stage in the specimen life would be dependent upon the number initially nucleated.

CONCLUSIONS

The results of experiments conducted in this project lead to the following conclusions.

1. For a prestress amplitude higher than the test stress amplitude:

(*a*) The damage at the test stress averaged about 4 times as great as the 5 per cent cycle ratio of prestress.

(*b*) More cycles of prestress did not cause a proportionately greater reduction in life at the test stress. The average per cent reduction at the test stress was about 2.3 times the 15 per cent cycle ratio of prestress, about 1.7 times the 35 per cent cycle ratio of prestress, and 1.1 times the 70 per cent cycle ratio prestress.

2. For a prestress amplitude lower than the test stress amplitude:

(*a*) Five per cent prestress cycle ratio generally resulted in an average life at the test stress 3 per cent greater than for a virgin specimen.

(*b*) Fifteen per cent cycle ratio generally caused further increases in life at the test stress, as high as 12 per cent improvement in one case.

(*c*) Thirty-five and 70 per cent prestress cycle ratios reduced the life significantly at the test stress—12.8 and 46.3 per cent respectively.

The above observations are consistent with a hypothesis that initial stress cycles modify the rate of damage accumulation from subsequent cycles of stress in the following way: A characteristic number of crack nuclei are initiated by the first cycles applied to the specimen, and further damage at other stress levels is confined primarily to propagation of these initial cracks. The number of cracks initiated is controlled by the initial stress level, and the rate at which they propagate is a function of the level of subsequent stress amplitudes.

Acknowledgments:

This paper is based on research supported by the Michigan College of Mining and Technology of which Frank Kerekes is dean. The authors wish to express their gratitude to Mr. T. Dimoff and Mr. G. Wayne Brown for their assistance in the experimental phase of the investigation.

APPENDIX

STATISTICAL ANALYSIS OF FATIGUE TEST DATA

Statistics of the Finite Life Curve:

The expression for the arithmetic mean of the logarithms is

$$\overline{\log N} = \sum_{i=1}^{i=n} \frac{\log N_i}{n}$$

Here N_i represents the fatigue life of the ith specimen and n is the number of tests conducted at a given stress level.

TABLE IV.—OBSERVATIONS TO ILLUSTRATE THE DETERMINATION OF MEAN OF TRUNCATED DATA.

Log N_k	k	P_k
[a]	1	9.1
3.38	2	18.2
3.65	3	27.3
3.65	4	36.4
3.73	5	45.5
3.97	6	54.6
4.06	7	63.6
4.09	8	72.7
4.13	9	81.8
4.22	10	90.9

[a] Failure during prestress.

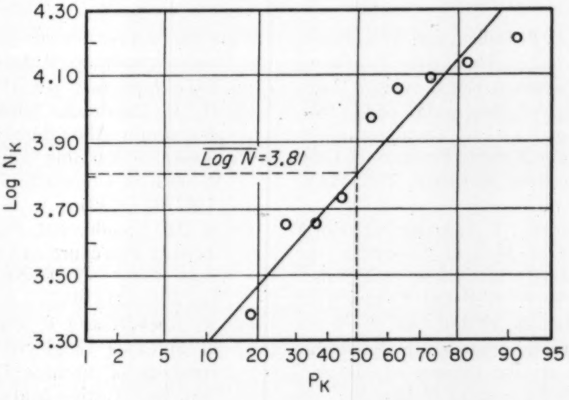


FIG. 9.—Graphical Determination of the Mean Life for Truncated Fatigue Data.

It is convenient to use logarithmic probability paper for the presentation of results at a fixed stress level, because on this paper the expected proportion P of specimens failing plot as a linear function of log N, the logarithm of the number of cycles (10). A value of P is obtained in the following manner for each specimen that fails: If n is the number of specimens tested and the k^{th} failure occurs at N_k cycles, then $P_k = \frac{k}{n+1}$ gives the proportion, P_k, of failure to be plotted at N_k cycles. Plotting log N on probability paper is equivalent to plotting N on logarithmic probability paper. The straight line drawn through the points is obtained by calculating the mean and the standard deviation of the log N values by the equation given previously. For a normal distribution, the mean and the median coincide; the mean of the log N values gives the point where the line intersects the 50 per cent failure line of the probability paper.

It is also possible to reverse the procedure and draw the line by eye and then use the fitted line to estimate the mean life.

In cumulative fatigue damage study the occurrence of failures during prestressing creates a problem in data analysis. Bennett (20) handled the problem by passing the S-N curve through the median of all the test points, thereby allowing inclusion of the specimens that failed at the prestress level.

Table IV and Fig. 9 illustrate the graphical determination of the mean life for fatigue data truncated by failure during prestressing. Results given are for tests in which a 70 per cent cycle ratio of prestress B was followed by cycling to failure at test stress A.

Determination of the Endurance Limit:

Each fatigue specimen has its own endurance limit. It has been shown by Ransom and Mehl (21) that the endurance limit is statistical in nature. Unfortunately, it is impossible to determine the endurance limit for a single specimen. If a specimen is tested at a fixed stress, the only information obtained regarding the endurance limit is whether the test stress is above or below the endurance limit for that specimen. As in the fatigue fracture portion of the *S-N* diagram, the information usually required is the mean endurance limit and a measure of the scatter. One method commonly employed is the "up-and-down," or "staircase" method.

In the "up-and-down" method, the stress used to test each specimen is based on the results of the preceding test. The stress at which the first specimen is tested is chosen as close as possible to the expected median endurance limit. An increment of stress called the step is also chosen. If the first specimen fails, the stress is decreased by one step for the second specimen; if the first specimen survives, the stress is increased by one step for the second specimen. Because of the time required to test the required number of specimens in sequence, it is convenient to follow separate up-and-down sequences on several machines and eventually combine the machine averages. A step size equal to the standard deviation of the distribution is the optimum choice, but because this number is not known in advance it is necessary to guess at an appropriate step size. A value anywhere from $\sigma/2$ to $2\ \sigma$ is satisfactory. Values of the standard deviation, σ, may be expected to range from 1500 psi for normalized or hot-rolled steel to 5000 psi for steel quenched and tempered to high hardness levels. The method of analysis of the data is outlined by Roeloffs and Garofalo (10).

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DISCUSSION

MR. PARKS TOOLIN.[1]—I would like to know if in the first stage the damage is really zero, or if under some circumstances in this strain aging material it could be a beneficial region and perhaps in other materials a region of lower slope but still a detrimental damage region.

Also, perhaps we too frequently confine our study of damage to laboratory specimens with multiple cracking. Many service failures start at stress concentrations and the probability of multiple cracking is less.

MR. C. E. WORK (*author*).—The two points mentioned by Mr. Toolin can be answered separately. The notion of zero damage is a difficult one to justify. It appears that whether initiation of microscopic cracks represents positive, negative, or zero damage depends on what is done to the material after the cracks are formed.

From the viewpoint of the designer, the warning that we restrict ourselves too often to laboratory specimens is valid. However, we are not in a position to evaluate as many different shapes as might be of interest. I would expect the initiation of many microcracks in service, even under severe stress concentrations. We have not investigated service failures in our studies.

MR. FRANK YURKOSKI.[2]—Realizing that laboratory specimens do not supply all the answers, I feel that they are beneficial to a certain extent. However, I wondered why specimens have not been tested that are free from highly polished surfaces. Possibly testing this material, or in fact any structural alloy, with as-rolled surfaces on portions of the specimen might be more beneficial.

Does Mr. Work feel that more correct values would have been obtained if he had selected this technique?

MR. WORK.—My opinion is that use of specimens with uncontrolled surface irregularities would have resulted not only in decreased life but also in increased scatter. It would probably have increased the number of specimens required at each level to obtain reasonable statistical validity. It seems likely that the amount of extra work required would not result in a corresponding increase in what we would learn about the accumulation of damage.

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[2] Metallurgist, Research and Development Division, ACF Industries, Inc., Berwick, Pa.

MR. YURKOSKI.—In the second portion of the paper, reference is made to the rate of crack propagation after initiation. Were the metallurgical characteristics of the crack studied with an electron microscope to determine at what stage in the life of the specimen the crack was initiated? Actually, what caused the crack to initiate?

MR. WORK.—The investigation reported here did not include any metallographic studies. Reference was made to the findings of others. It would be desirable to extend this investigation to metallographic confirmation of the speculations made concerning initiation and propagation of cracks.

EFFECT OF LOADING SEQUENCE ON CUMULATIVE FATIGUE DAMAGE OF 7075-T6 ALUMINUM ALLOY*

BY ROBERT SPITZER[1] AND H. T. CORTEN[1]

SYNOPSIS

For conditions of fluctuating stress amplitude consisting of repeated blocks of cycles of two stress amplitudes and continuously varying amplitudes, it has been shown that the mean fatigue life may be predicted using the hypothesis of linear summation of cycle ratios and a modified σ-N relation. This approach assumes that the initial sequence of high, intermediate, and low stresses does not significantly influence the fatigue life, since, in general, the number of cycles in each repeated block is small compared to the life of the member. Recent studies by Naumann and Hardrath suggest, however, that the sequence of applying high, intermediate, and low stresses in repeated-block experiments may change the fatigue life by a factor of from 2 to 4. This investigation was undertaken to clarify this question. It was found that changing the sequence from high followed by low stresses to low followed by high stresses in each repeated block did not change the life by more than the length of one repeated block of cycles, or by a factor of from 1.07 to 1.14.

To meet competition and increase performance, machines and structures of all varieties—from farm equipment to aircraft and from roller chain to missiles—contain components that are designed on a limited fatigue life basis. The members experience variable-amplitude stresses that reflect first the functional operation of the member and, second, transient and random vibratory stresses superimposed upon the operational load patterns. Regardless of origin, the resulting stress patterns frequently are complex, containing a few cycles of peak stress, a larger number of cycles of intermediate stress, and a very large number of cycles of low stress, all mixed randomly together.

The earliest attempt to estimate the fatigue life of a member subjected to variable-amplitude loading was based on the hypothesis that the fatigue life was "consumed" gradually (**1–3**);[2] each cycle of stress σ_i consumed the life of the member in proportion to the ratio $1/N_i$, where N_i is the life under constant-amplitude stress σ_i. If n_i cycles were applied at stress σ_i, the cycle ratio n_i/N_i represented the proportion of the life of the member consumed. For a member that encountered n_1 cycles of stress σ_1, n_2 cycles of stress σ_2, $\cdots$ n_i cycles of stress σ_i, etc., the total life of the member presumably was consumed when

$$n_1/N_1 + n_2/N_2 + \cdots + n_i/N_i = 1 \quad (1)$$

where N_1, N_2, $\cdots$ N_i are the fatigue lives from constant-stress-amplitude experiments at stresses σ_1, σ_2, $\cdots$ σ_i, respectively.

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* Presented at the Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] University of Illinois, Urbana, Ill.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

Equation 1, which states the hypothesis usually called the linear summation of cycle ratios, assumes that each cycle of stress consumes the life of the member by an amount that is entirely independent of previous or subsequent cycles of stress. For many years data have been accumulated which indicate that this assumption is an oversimplification of the actual situation (4).

Recent investigations have shown that a modification of the linear-summation hypothesis provides a simple relation of greatly improved accuracy (5–8). In this modification, Eq 1 is used with a modified σ-N relation. In Fig. 1, the conventional σ-N relation is represented by the solid line *ab*, and two possible modified σ-N relations are represented by the dashed lines *ac* and *ad*. Used in conjunction with the appropriate modified σ-N relation, it has been shown that Eq 1 gives excellent agreement with a large amount of experimental data from experiments that were planned and analyzed by statistical methods. The basis for the use of the modified σ-N relation will be discussed in detail in the next section of this paper.

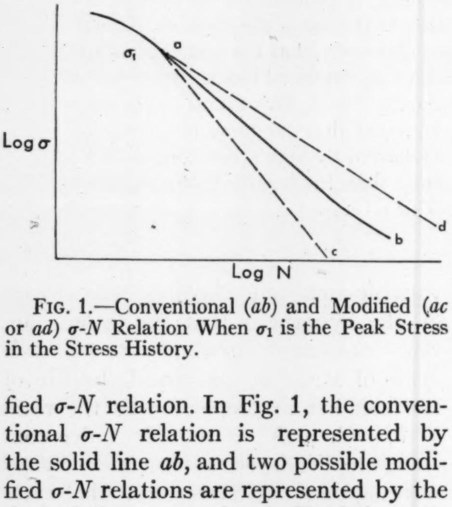

FIG. 1.—Conventional (*ab*) and Modified (*ac* or *ad*) σ-N Relation When σ_1 is the Peak Stress in the Stress History.

In many recent studies, repeated patterns of stress cycles have been used to reduce the influence of the statistical variability of fatigue life on the results. That is, if the length of each repeated block of cycles was short compared to the total life of the specimen, each specimen, regardless of its life, would experience the same proportion of cycles at high, intermediate, and low stresses. In addition, it was assumed that with short repeated blocks of cycles relative to the total life, the order of applying the high, intermediate, and low cycles of stress within each repeated block would have a small, possibly negligible, influence on the life.

Recent data by Hardrath and Naumann (9) and also Schijve (10) indicate, however, that within each repeated block of cycles the sequence of applying the high, intermediate, and low stress cycles may influence the fatigue life by a factor of as much as 4. These two investigations, however, showed conflicting trends with regard to the influence of sequence of stresses within each repeated block. Hardrath and Naumann found that the life was shortest when the low stresses in each repeated block were applied first; conversely, Schijve found that the life was shortest when the high stresses were applied first. These differences suggest that either the results were not statistically significant or that influences unknown to the investigators were present in their data.

This investigation was made to determine the influence on fatigue life of the sequence of applying the low and high stresses within each repeated block of cycles. This work was an extension of previous work using the same material, machine, and method of analysis. Thus, considerable data from more than 2300 specimens were available for comparison. In previous work two types of stress histories were used: two-stress, repeated-block histories (Fig. 2), and continuously-varying-stress, repeated-block histories (Fig. 3). In earlier work, each experiment was started just after the highest-stress portion of the cycles was

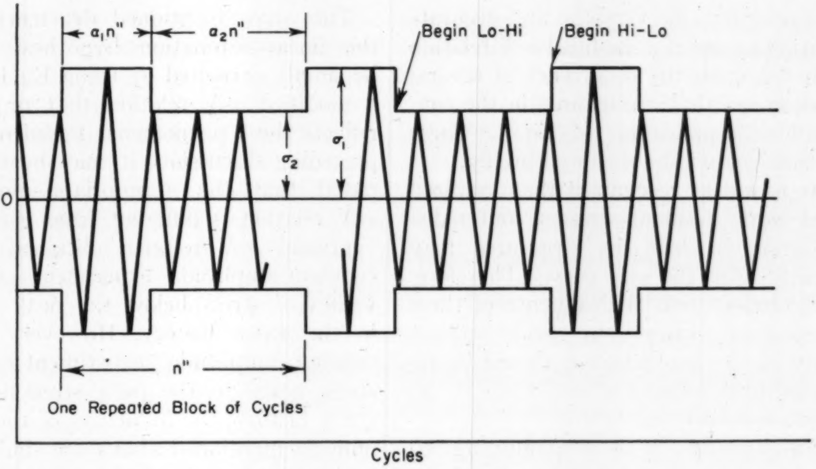
$a_1 n''$
$a_2 n''$
Begin Lo-Hi
Begin Hi-Lo
σ_1
σ_2
Stress
0
n"
One Repeated Block of Cycles
Cycles

FIG. 2.—Two-Stress, Repeated-Block Stress History.

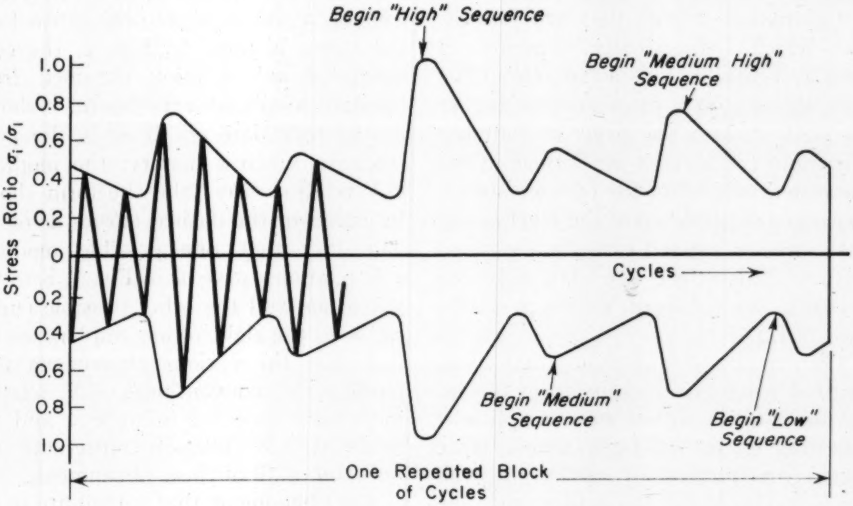
Begin "High" Sequence
Begin "Medium High" Sequence
1.0
0.8
0.6
0.4
0.2
0
0.2
0.4
0.6
0.8
1.0
Stress Ratio σ_i/σ_1
Cycles ⟶
Begin "Medium" Sequence
Begin "Low" Sequence
One Repeated Block of Cycles

FIG. 3.—Continuously Varying Stress History.

completed. Thus, if there was an influence of sequence of applying the high and low stresses, it probably was not evident in the earlier data, but it could lead to an over-optimistic view of the reproducibility of the data.

In this investigation, the data will be interpreted in terms of the modified σ-N relation and Eq 1. Therefore the concept of a modified σ-N relation will be reviewed briefly.

CUMULATIVE FATIGUE DAMAGE AND THE MODIFIED σ-N RELATION

The concept of linear accumulation of damage expressed by Eq 1, used in conjunction with a modified σ-N relation, has proved to be versatile and accurate (**7,11**). The use of a modified σ-N relation is based upon the occurrence of several phenomena that are ignored in the conventional application of Eq 1. These phenomena will be reviewed briefly.

It is widely recognized that localized cold work, residual stresses, and redistribution of load in structures may greatly alter the σ-N curve. Therefore, it is obvious that the influence of these phenomena, caused by peak stresses early in the load history, should be reflected in the values of $N_1, N_2, \cdots N_i$ that are used in Eq 1.

Furthermore, it is generally agreed that cracks may nucleate early in the life of a member subjected to variable-amplitude loading. Frequently the number of minute cracks that are formed, and which subsequently combine to form a visible crack, is a function of the peak stresses on a member. The higher the peak stresses, the larger the number of minute cracks that are formed. Thus the rate at which the life of a member is consumed depends upon the level of the peak stresses applied early in the stress history. This effect also should be reflected in the values of $N_1, N_2, \cdots N_i$ used in Eq 1.

The final phenomenon that was omitted from Eq 1 results from the influence of cycles at low stress amplitude[3] following cycles of peak stress. Once cracks are formed by cycles of peak stress, cycles of low stress propagate the cracks and consume fatigue life. A large number of cycles of stress just below the fatigue limit can consume an appreciable amount of the fatigue life of a member. This effect should be included by extending the σ-N relation below the conventional fatigue limit.

The above-mentioned deficiencies in the linear-summation hypothesis may be simply corrected by using Eq 1 with a modified σ-N relation that properly reflects these phenomena. Based on the preceding discussion, it may be anticipated that the appropriate modified σ-N relation is different from the conventional σ-N relation obtained from constant-amplitude experiments at all values of stress below the peak stress in the stress history. However, for a constant-amplitude experiment at a stress equal to the peak stress in the stress history, the influences of the phenomena mentioned above are similar in both the constant- and variable-amplitude stress histories. Therefore the modified and conventional σ-N relations coincide at the value of peak stress from the stress history. In Fig. 1, the conventional σ-N relation obtained from constant-amplitude experiments is shown as the solid line *ab*. If σ_1 is the peak stress in a stress history, the modified σ-N relation may take the form shown by either of the dashed curves *ac* or *ad*. The fact that one possible modified σ-N relation, curve *ac* in Fig. 1, is to the left of *ab*, and the other possible curve, *ad*, is to the right of *ab*, emphasizes the fact that the various phenomena that modify the conventional σ-N relation may have opposing influences, and the modified σ-N relation represents the *resultant* of all of these phenomena.

The phenomena that contribute to the modified σ-N relation are complex, and the appropriate relation is difficult to predict quantitatively. However, it is frequently observed that for small unnotched rotating-beam specimens, curve *ac* in Fig. 1 would be the appropriate modified σ-N relation. In axial-load experiments, the introduction of a mean stress or a notch or both tends to shift the modified σ-N relation to the right, and in some instances it may approximately coincide with *ab*. For structural components containing riveted joints or full-size structures subjected to both mean and alternating stresses, the modified σ-N relation shifts still further to the right, toward curve *ad*. The question of obtaining an appropriate modified σ-N relation will be discussed later.

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[3] Cycles of low stress in this instance refer to cycles of stress below the fatigue strength (10^7 cycles) as determined from constant-amplitude experiments.

Assuming for a moment that an approximate modified σ-N relation is available, the number of cycles to fracture at stresses $\sigma_1, \sigma_2, \cdots \sigma_i$, will be denoted by N_1'; $N_2', \cdots N_i'$, respectively. Using the modified σ-N relation, Eq 1 becomes

$$\frac{n_1}{N_1'} + \frac{n_2}{N_2'} + \cdots + \frac{n_i}{N_i'} = \sum_{i=1}^{m} \frac{n_i}{N_i'} = 1 \ldots (2)$$

To estimate the life of a member subjected to cycles of stress at variable amplitude, it is convenient to redefine the quantities in Eq 2 in terms of the variables that appear in the load or stress spectrum. If N_g is the number of cycles to failure in a variable-stress-amplitude experiment, the number of cycles n_1 at the peak stress σ_1 will be denoted by $\alpha_1 N_g$. Similarly, $n_2 = \alpha_2 N_g, \cdots n_i = \alpha_i N_g$. Making these substitutions in Eq 2, the resulting expression can be solved for the fatigue life N_g, giving

$$N_g = \frac{1}{\dfrac{\alpha_1}{N_1'} + \dfrac{\alpha_2}{N_2'} + \cdots + \dfrac{\alpha_i}{N_i'}}$$

$$= \frac{N_1'}{\alpha_1 + \alpha_2 \dfrac{N_1'}{N_2'} + \cdots + \alpha_i \dfrac{N_1'}{N_i'}}$$

Finally,

$$N_g = \frac{N_1'}{\sum\limits_{i=1}^{m} \alpha_i \left(\dfrac{N_1'}{N_i'}\right)} \ldots\ldots\ldots (3)$$

However, since σ_1 was defined as the peak stress, $N_1 = N_1'$.

For many materials it has been found that the portion of the σ-N relation that is of interest can be adequately represented by a straight line on a log-log diagram. When this is the case, the conventional σ-N relation can be represented by a relation of the form

$$\log \sigma_i = A - \left(\frac{1}{\delta}\right) \log N_i \ldots\ldots (4)$$

where A is the intercept of this line at $N = 1$, and $\left(\dfrac{1}{\delta}\right)$ is the slope of the line. Similarly, the modified σ-N relation is given by

$$\log \sigma_i = A' - \frac{1}{d} \log N_i' \ldots\ldots. (5)$$

The intercept A' is adjusted so that the two lines, Eqs 4 and 5, intersect at the point σ_1, N_1; however, the modified σ-N relation is not defined for stresses larger than σ_1, the peak stress in the stress history. The slope $\dfrac{1}{d}$ may be larger than, equal to, or smaller than $\dfrac{1}{\delta}$, as discussed previously.

Using Eq 5, the ratio $\dfrac{N_1'}{N_i'}$ appearing in Eq 3 may be expressed in terms of the stresses $\sigma_1, \sigma_2, \cdots \sigma_i$, as

$$\left(\frac{\sigma_2}{\sigma_1}\right)^d = \frac{N_1'}{N_2'}; \left(\frac{\sigma_i}{\sigma_1}\right)^d = \frac{N_1'}{N_i'}$$

Upon substitution into Eq 3, the expression for the fatigue life N_g becomes

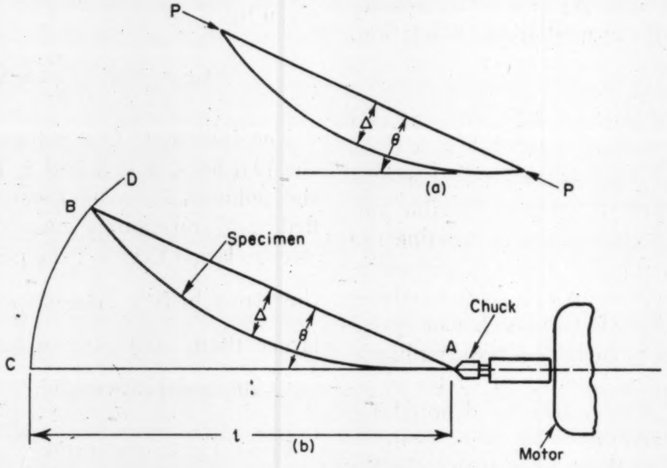
$$N_g = \frac{N_1}{\alpha_1 + \alpha_2 \left(\dfrac{\sigma_2}{\sigma_1}\right)^d + \cdots + \alpha_i \left(\dfrac{\sigma_i}{\sigma_1}\right)^d}$$

$$= \frac{N_1}{\sum\limits_{i=1}^{m} \alpha_i \left(\dfrac{\sigma_i}{\sigma_1}\right)^d} \ldots (6)$$

Equation 6 is a convenient form for use in computing fatigue life N_g. In Eq 6, the modified σ-N relation is represented by the parameter d. If $d = \delta$, the modified σ-N relation coincides with the conventional σ-N relation and Eq 6 is equivalent to Eq 1, but arranged for convenient computation of N_g. If $d > \delta$, the modified σ-N relation corresponds to line *ad* in Fig. 1, and, conversely, if $d < \delta$, the modified σ-N relation corresponds to line *ac*.

It is interesting to note that Eq 6 contains one experimental parameter, d, in addition to those contained in Eq 1. Considering the deficiencies of Eq 1, Eq 6 represents one of the simplest forms of equation that could represent the data more accurately than Eq 1.

Returning to the question of the influence of the sequence of applying loads, it is clear that, for simplicity, it would be highly desirable to be able to ignore the sequence of cycles of stress in a repeated block. Because it is impossible to predict the sequence of loads in many service applications, an influence of stress sequence within a repeated block simply means that the accuracy of a fatigue life estimate is poor.



(*a*) Free body diagram of the wire specimen.
(*b*) Layout of the wire machine.

FIG. 4.—Schematic Diagram of Wire Machine.

EXPERIMENTAL INVESTIGATION

In this study, 7075-T6 aluminum alloy wire specimens were used (6,7). The yield strength at 0.2 per cent offset was 69,000 psi, and the ultimate strength was 80,000 psi.

The wire specimens were loaded as a deflected rotating strut as shown schematically in Fig. 4(*a*). Rotation of the specimens at speeds between 7000 and 8000 rpm caused completely reversed cycles of stress,[4] with the maximum stress at the center of the specimen length. The maximum alternating stress was computed using the relation (12):

$$\sigma = \left(\frac{Pl^2}{EI}\right)\left(\frac{\Delta}{l}\right)\left(\frac{Ec}{l}\right) \qquad \text{........(7)}$$

where:

P = axial compressive load, lb,
l = specimen length, in.,
E = modulus of elasticity, psi,

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[4] The axial compressive steady stress was less than 1 per cent of the alternating bending stress, and was ignored.

I = moment of inertia of cross-section, in.^{4},

Δ = maximum deflection at the specimen midpoint, in., and

c = radius of cross-section, in.

TABLE I.—FATIGUE DATA.

High Stress, σ_1, psi	Low Stress, σ, psi	Per Cent of Life at High Stress, α	Stress Sequence	Log N_g		Standard Deviation	N_g (antilog of mean log N_g)
				Mean	±95 Per Cent Confidence Limits		
CONSTANT-STRESS-AMPLITUDE							
50 000	...	100	...	4.2544	0.0329	0.0685	18 000
30 000	...	100	...	5.2900	0.0368	0.0766	195 000
50 000	...	100	...	4.2720	0.0132	0.0275	18 700
TWO-STRESS, REPEATED-BLOCK STRESS HISTORY							
50 000	40 000	4	Hi-Lo	4.6485	0.0162	0.0337	44 500
		4	Lo-Hi	4.6900	0.0218	0.0455	49 000
50 000	40 000	10	Hi-Lo	4.6390	0.0237	0.0494	43 600
		10	Lo-Hi	4.6779	0.0203	0.0422	47 600
50 000	30 000	4	Hi-Lo	5.2320	0.0208	0.0433	170 600
		4	Lo-Hi	5.2856	0.0323	0.0674	193 000
50 000	30 000	10	Hi-Lo	5.0176	0.0211	0.0439	104 100
		10	Lo-Hi	5.0509	0.0170	0.0353	112 400
50 000	20 000	4	Hi-Lo	5.5985	0.0145	0.0303	396 700
		4	Lo-Hi	5.6268	0.0140	0.0293	423 500
50 000	20 000	10	Hi-Lo	5.1929	0.0059	0.0123	155 900
		10	Lo-Hi	5.2296	0.0143	0.0299	169 700
CONTINUOUSLY VARYING STRESS HISTORY							
50 000	15 000	...	high	5.3836	0.0167	0.0349	241 900
		...	medium-high	5.3960	0.0202	0.0421	248 900
		...	medium	5.3762	0.0181	0.0378	237 800
		...	low	5.3926	0.0162	0.0337	247 000

Referring to Fig. 4, the dimensionless ratios $\frac{Pl^2}{EI}$ and $\frac{\Delta}{l}$ are functions of the angle θ only. The quantity $\frac{Ec}{l}$ is a constant for a particular material and geometry.

The stress amplitude was varied by shifting the specimen end B along the path CD. Path CD was constructed to produce approximately zero bending moment at each end of the specimen (12). For the two-stress, repeated-block history shown in Fig. 2, the specimen end B was shifted between two predetermined positions on the path CD by a solenoid controlled by a synchronized cam. The continuously varying stress history shown in Fig. 3 was obtained by attaching specimen end B to a contour cam through a cam follower. The stress patterns shown in Figs. 2 and 3 were repeated every 10,000 cycles.

The wire, which was received in straightened 3-ft lengths, was cut to length, cleaned with acetone, and coated with a thin layer of petroleum jelly to establish a reproducible surface environment for all specimens (7). The specimens were tested in the as-drawn surface condition.

For the two-stress, repeated-block history, a high stress (σ_1) of 50,000 psi and three values of low stress (σ_2) of 20,000, 30,000, and 40,000 psi were used. For each of the three combinations of high and low stress, two values of the fraction of cycles at the high stress, $\alpha = 0.04$ and $\alpha = 0.10$, were used. Thus six combinations of conditions were used to investigate the influence of sequence of low and high stresses. In Fig. 2 the sequence denoted Lo-Hi started at the beginning of the low stress cycles in the interval; similarly the Hi-Lo sequence started at the beginning of the high stress interval. Twelve sets of data of 20 specimens each were obtained.

For the continuously varying stress history shown in Fig. 3, the peak stress in the history was chosen as 50,000 psi, and the minimum amplitude of stress was 0.3 of the peak, or 15,000 psi. One revolution of the contour cam was divided into 200 parts and calibrated to establish the curve shown in Fig. 3. For this load history, four starting points were selected, denoted by high, medium-high, medium, and low in Fig. 3. For each starting point, 20 specimens were used.

A logarithmic-normal frequency distribution was assumed in the analysis of all data. The experimental conditions are given in the first four columns of Table I, and the results of the statistical analysis of the fatigue data are given in the next three columns. The last column is the value of fatigue life N_g in cycles obtained from mean log N_g.

From inspection of Table I, it is clear that there was a difference between the Hi-Lo and Lo-Hi sequence for the two-stress, repeated-block experiments. The Hi-Lo sequence consistently led to the shorter life but the difference was small. The ratio of the longest life to the shortest life ranged from 1.07 to 1.14. Based on 95 per cent confidence limits, some of the values of life for the Hi-Lo sequence overlapped the lives for the Lo-Hi sequence, while others did not. In the Lo-Hi sequence, if it is assumed that no damage is done by the cycles of low stress that precede the first high-stress cycles, it would be reasonable to expect that the life of the Lo-Hi sequence would be nearly one block of cycles (9000 to 9600 cycles) longer than the Hi-Lo sequence. The absolute differences in fatigue life range from approximately $\frac{1}{2}$ to 2 repeated blocks of cycles.

For the continuously varying stress histories, the fatigue lives for the four sets of data are very similar. The longest one is less than 5 per cent longer than the shortest one. The 95 per cent confidence limits of all four sets of data overlap, indicating that the differences in the fatigue lives are not statistically significant at this level.

DISCUSSION OF RESULTS

The results presented in Table I provide a direct answer to the question of the influence on fatigue life of the sequence of loading within each repeated block. However, this question is only one phase of the problem of estimating the fatigue life of members and structures. The larger problem is how to determine the appropriate modified σ-N relation. The concept of a modified σ-N relation as outlined earlier contains the assumption that this relation is unique for a particular specimen configuration and peak stress. If this is so, then the same modified σ-N relation will be obtained regardless of the nature of the load spectrum below the peak stress. In practical terms this means that the simplest stress history—the two-stress, repeated-block history—is adequate to determine the value of d in Eq 6 for use with any complex loading condition. Since a small influence of stress sequence was observed, it appears desirable to examine the error introduced in the computed fatigue life for conditions of continuously varying stress amplitude.

This question will be taken up in two phases: (1) the determination of values of d from the two-stress, repeated-block stress histories, and (2) computation of the fatigue life N_g for the continuously varying stress history using those values of d.

To evaluate d, the inverse slope of the modified σ-N relation from the two-stress, repeated-block experiments, Eq 6, may be simplified to

$$\frac{N_g}{N_1} = \frac{1}{\alpha_1 + \alpha_2 \left(\frac{\sigma_2}{\sigma_1}\right)^d} \qquad (8)$$

It is convenient to replace the ratio $\left(\frac{\sigma_2}{\sigma_1}\right)^d$ by the symbol $(R^{1/a})$ for experimental evaluation.[5] Solving for $R^{1/a}$ gives

$$R^{1/a} = \frac{1}{\left(\frac{N_g}{N_1}\right)\alpha_2} - \frac{\alpha_1}{\alpha_2} \qquad (9)$$

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[5] The quantity $(R^{1/a})$ is used simply to maintain consistency of symbols with previous publications (**5–8**).

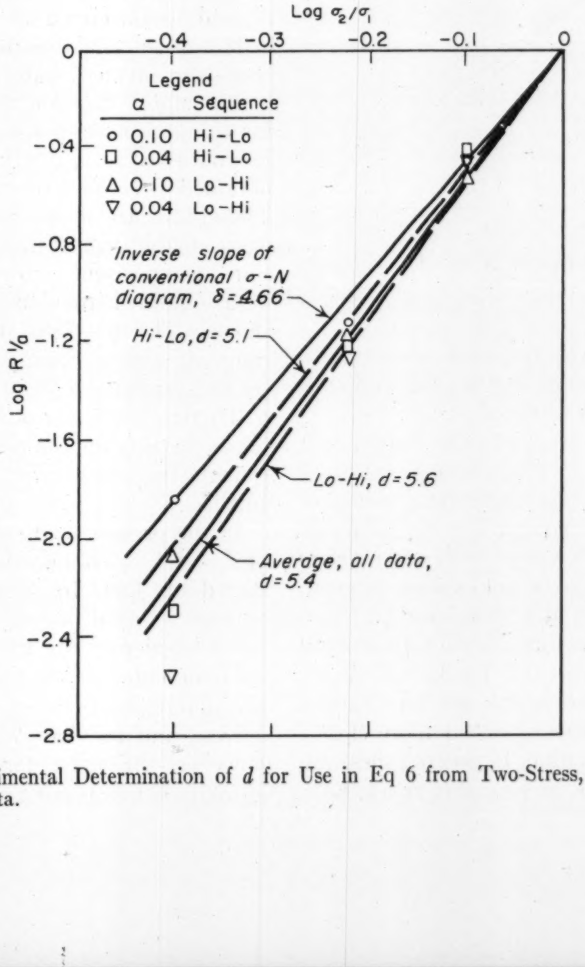

FIG. 5.—Experimental Determination of d for Use in Eq 6 from Two-Stress, Repeated-Block Stress History Data.

From experimental values of α_1, α_2, and $\frac{N_g}{N_1}$, values of $R^{1/a}$ were computed from Eq 9 for each experimental condition. The value of d was determined from the relation

$$R^{1/a} = \left(\frac{\sigma_2}{\sigma_1}\right)^d$$

First, the logarithm was taken of both sides of Eq 10, giving

$$\log (R^{1/a}) = d \log (\sigma_2/\sigma_1) \; \ldots\ldots \quad (11)$$

It was then noted that d is the slope of the line through the data points in a diagram with log $R^{1/a}$ as the ordinate and log (σ_2/σ_1) as the abscissa.

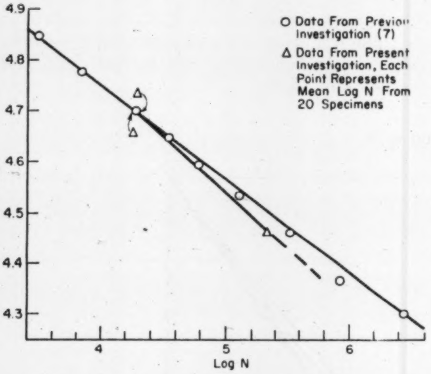

FIG. 6.—σ-N Diagram for 7075-T6 Aluminum Alloy Wire.

In Fig. 5, experimentally determined values of log $R^{1/a}$ computed from Eq 9 are shown for the appropriate values of log (σ_2/σ_1). In computing the values of $R^{1/a}$ from Eq 9, values of α_1 and α_2 were computed from the final values of mean log N_g. Since the mean fatigue lives usually did not coincide with an integral number of repeated blocks of cycles, the nominal values of α_i given in Table I were adjusted to represent more closely the actual situation. In general, these adjustments of α_1 and α_2 were small; however, they tended to reduce the scatter of data in Fig. 5.

In Fig. 5, the data exhibit surprisingly little scatter for σ_2/σ_1 of 0.8 and 0.6, but the scatter is greater at 0.4. This tendency has been observed previously (7) and tends to indicate that if only a few sets of data are available, greater accuracy in the determination of d is obtained for a stress ratio in the neighborhood of 0.6.

Four lines have been drawn in Fig. 5. The two dashed lines represent the best fit to the Lo-Hi data and the Hi-Lo data. The solid line between the dashed lines is the line of best fit to all of the data. These results indicate that the value of d is sensitive to the influence of sequence of stressing. The influence of this variation of d on the estimate of fatigue life N_g will be considered later.

The other solid line in Fig. 5 represents the inverse slope, δ, of the conventional σ-N diagram and was included for comparison with the data. This line was obtained from the three sets of data obtained at a constant stress amplitude. These data are shown in Fig. 6 plotted on a double logarithmic σ-N diagram. For comparison, constant-amplitude data obtained previously (7) are also shown. The previous data covered a range of stresses from 20,000 to 70,000 psi and exhibited a linear relation in Fig. 6. Therefore it is reasonable to expect a linear relation for the present data even though the number of data points is small.

In the present work an average value of $d = 5.4$ was obtained, and δ was estimated at 4.66. In previous work the average value of d was 5.8, and δ was 5.4. Considering the uncertainty in the value of δ obtained in the current work, the differences between d and δ in the current and previous work are similar; however, the cause for the change in absolute values is not known.

The value of N_g was computed using Eq 6. To evaluate the denominator of Eq 6, the continuously varying stress history was divided into 20 equal stress increments and the number of cycles in each increment was counted. To illustrate the stress spectrum, the number of stress intervals was reduced to 10 as shown in Fig. 7. The fraction of the cycles in each interval was plotted as the abscissa on a logarithmic scale, and the stress ratio σ_i/σ_1 was plotted as the ordinate. Frequently, stress spectrum diagrams of this type produce a nearly linear variation of log α with σ_i/σ_1. The particular cam used in this study caused a relatively large number of cycles to occur in the peak stress interval. Previous work (7) indicates, however, that the fatigue lives are not influenced in any unusual manner by the large value of α in the peak stress interval.

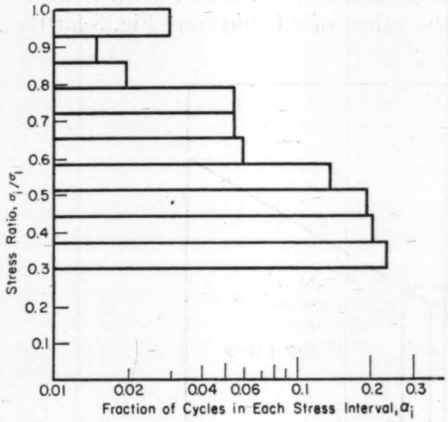

FIG. 7.—Stress Spectrum for Continuously Varying Stress History.

The computation of fatigue life N_g is illustrated in Table II. The stress intervals are shown in the first column and the mean stress ratio for each interval

TABLE II.—COMPUTATION OF FATIGUE LIFE N_g FOR CONTINUOUSLY VARYING STRESS HISTORY USING EQ 6.[a]

Stress Ratio Increments	$\frac{\sigma_i}{\sigma_1}$	$\left(\frac{\sigma_i}{\sigma_1}\right)^d$	α_i	$\alpha\left(\frac{\sigma_i}{\sigma_1}\right)^d$
1.00 to 0.966	0.9825	0.9090	0.020	0.0182
0.965 to 0.931	0.9475	0.7474	0.010	0.0075
0.930 to 0.896	0.9125	0.6099	0.010	0.0061
0.895 to 0.861	0.8775	0.4938	0.005	0.0025
0.860 to 0.826	0.8425	0.3964	0.005	0.0020
0.825 to 0.791	0.8075	0.3152	0.015	0.0047
0.791 to 0.756	0.7725	0.2481	0.030	0.0074
0.755 to 0.721	0.7375	0.1932	0.025	0.0048
0.720 to 0.686	0.7025	0.1486	0.025	0.0037
0.685 to 0.651	0.6675	0.1127	0.030	0.0034
0.650 to 0.616	0.6325	0.0843	0.030	0.0025
0.615 to 0.581	0.5975	0.0620	0.030	0.0019
0.580 to 0.546	0.5625	0.0447	0.025	0.0011
0.546 to 0.511	0.5275	0.0316	0.110	0.0035
0.510 to 0.476	0.4925	0.0218	0.095	0.0021
0.475 to 0.441	0.4575	0.0185	0.100	0.0018
0.440 to 0.406	0.4225	0.0095	0.105	0.0010
0.405 to 0.371	0.3875	0.0060	0.095	0.0006
0.370 to 0.336	0.3525	0.0036	0.090	0.0003
0.335 to 0.300	0.3175	0.0020	0.145	0.0003

[a] $\Sigma\alpha_i\left(\frac{\sigma_i}{\sigma_1}\right)^d = 0.0754.$

$N_1 = 17{,}960$ cycles.

$d = 5.4.$

$$N_g = \frac{17{,}960}{0.0754} = 238{,}300 \text{ cycles.}$$

is listed in column two. A value of $d = 5.4$, the average value for all of the data, was used in the computation, and values of $(\sigma_i/\sigma_1)^d$ are listed in column three. The values of α obtained from counting the cycles in each stress interval are listed in column four, and the product $\alpha_i(\sigma_i/\sigma_1)^d$ in column five. Summation of column five gives the value of the denominator in Eq 6. The remaining computations for N_g are shown below Table II, resulting in a fatigue life, N_g, of 238,300 cycles. Comparison with the experimental values in Table I indicates excellent agreement. The maximum difference is less than 5 per cent. Note also the simplicity of the computations.

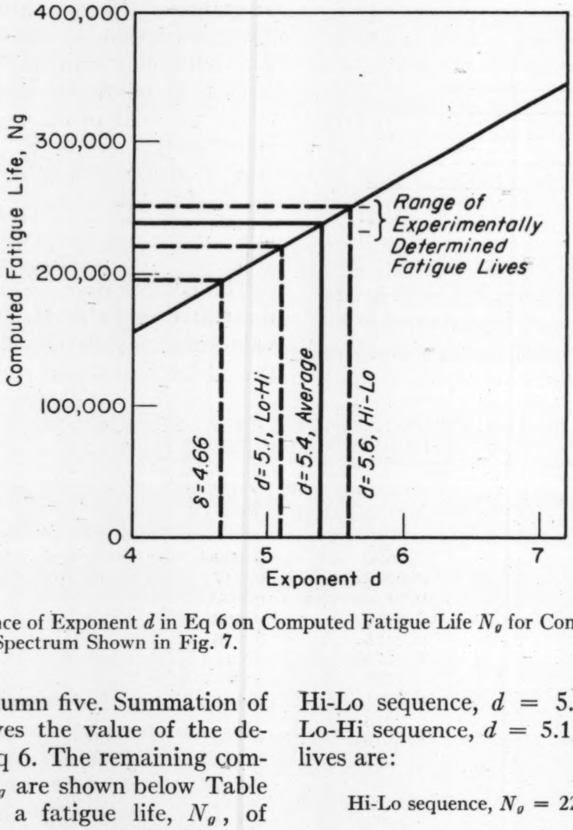

FIG. 8.—Influence of Exponent d in Eq 6 on Computed Fatigue Life N_g for Continuously Varying Stress Amplitude Spectrum Shown in Fig. 7.

The life computation discussed above was made with $d = 5.4$. The influence of the value of d on the life was investigated by computing the life for a series of values of d. The results of these computations are shown in Fig. 8. The life N_g is shown as the ordinate and d as the abscissa. The curve of N_g as a function of d is nearly linear and relatively flat. Thus N_g is relatively insensitive to small variations in the value of d. In terms of the values of d found from Fig. 5 for the Hi-Lo sequence, $d = 5.6$, and for the Lo-Hi sequence, $d = 5.1$, the computed lives are:

Hi-Lo sequence, $N_g = 220{,}000$ cycles

Lo-Hi sequence, $N_g = 250{,}000$ cycles

From these values of life, it is clear that the sequence of stressing in a two-stress, repeated-block experiment has a small influence on estimates of fatigue life; however, the ratio of N_g for $d = 5.1$ to N_g for $d = 5.6$ is only 1.14. Thus, from a practical point of view, the sequence of stresses appears to have a small, practically negligible influence. Other factors enumerated earlier produce differences of several hundred per cent. Until these other factors are better understood and controlled, it appears that the influence of sequence of stressing on fatigue life in repeated-block experiments may be disregarded.

CONCLUSIONS

The following conclusions appear justified:

1. In two-stress, repeated-block experiments, the influence of the sequence of stressing is present but it is small. For the stress sequences denoted Lo-Hi and Hi-Lo, the ratio of the fatigue lives (antilog of mean log N) varied from 1.07 to 1.14.

2. In the continuously varying stress history experiments, the sequence of stressing caused no measurable difference in fatigue life. From four sets of data, the ratio of the longest to shortest fatigue life was less than 1.05.

3. The fatigue life for continuously varying stress history experiments can be accurately estimated using Eq 6 and a value of d determined in two-stress, repeated-block experiments. Using the values of d obtained from the Hi-Lo sequence and the Lo-Hi sequence—two-stress, repeated-block experiments—a difference of 14 per cent between the two fatigue life estimates was obtained. The experimental values of fatigue life were bracketed between these two estimates.

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A RELATION BETWEEN THEORETICAL STRESS CONCENTRATION FACTOR AND FATIGUE NOTCH FACTOR DEDUCED FROM THE CONCEPT OF HIGHLY STRESSED VOLUME*

By R. Kuguel[1]

Synopsis

Size and shape effects in existing fatigue data are accounted for by a simple analysis based on the fact that a decrease in fatigue strength accompanies an increase in the volume of material subjected to at least 95 per cent of the maximum stress. A linear relation is established between the logarithm of the maximum stress and the logarithm of the appropriate volume, which is equally valid for smooth and notched members. Based on this analysis, the fatigue notch factor is expressed as a function of the theoretical stress concentration factor, the root radii of the notched and unnotched specimens, and the diameters of the specimens. This is proposed as a simple, adequate relation for use in design.

NOTATION

A	Area formed by the intersection of the highly stressed volume, V, with a plane containing the center line of the specimen, sq in.
a	Exponent
B	Coefficient depending on the material
C	Coefficient
b	Width of a plate, in.
d	Critical or smallest diameter of a specimen, in.
d_1	Diameter of a specimen at the extremes of the volume V, in.
d'	Distance between centroids of areas A, in.
F	Area of the cross-section of a plate, sq in.
G	Specific stress gradient, 1/in.
h	Height of the area A, in.
K_f	Fatigue notch factor
K_t	Theoretical stress concentration factor
M	Bending moment, in.-lb.
q	Notch sensitivity factor
r	Radius of the profile of a specimen, in.
S	Statistical standard deviation
t	Thickness of a plate, in.
V	Volume of a specimen subjected to 95 per cent and more of the maximum stress, cu in.
W	Section modulus, in^3.
α	$S_{\sigma_{fs}}/S_{\sigma_{fn}}$
Δl	Semibase of the triangle formed by the area A, in.
σ_f	The fatigue strength of a metal, psi (for aluminum alloys, the stress corresponding to 10^7 cycles)
$\sigma_{\max}$	Maximum stress, psi
σ_{nom}	Nominal stress, psi
σ_u	Ultimate strength, psi

Subscripts:

n	for notched specimens
s	for smooth specimens

It is well known that most metal members with stress raisers do not fully respond to the reduction of fatigue

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strength predicted by the "theoretical stress concentration factor" K_t as defined by

$$K_t = \frac{\sigma_{\max}}{\sigma_{\text{nom}}} \qquad (1)$$

where $\sigma_{\max}$ is the maximum elastic stress caused by the stress raiser and σ_{nom} is the nominal stress computed from the ordinary formulas of strength of materials at the same cross-section. For this reason an empirical factor K_f, the "fatigue notch factor," given by:

$$K_f = \frac{\sigma_{fs}}{\sigma_{fn}} \qquad (2)$$

should be used in design. In Eq 2, σ_{fs} and σ_{fn} are the fatigue strengths for smooth and notched specimens, respectively. Except for a few very dubious cases, the inequality $K_f < K_t$ is observed experimentally. Among the explanations that have been advanced regarding this inequality, the one based on inelastic deformation at the root of the notch is the most popular. Nevertheless, it does not fully and consistently explain the phenomenon.

The important question that arises in design is the prediction of the appropriate value of K_f. Here, the important concept of a "notch sensitivity factor," q, as defined by:

$$q = \frac{K_f - 1}{K_t - 1} \qquad (3)$$

was introduced by Peterson (**1**).[2] The q factor has the merit of providing a scale for the notch sensitivity that varies between $q = 0$ (no notch effect) and $q = 1$ (full theoretical effect). Peterson suggested that q depends upon the radius of the notch r_n and the hardness of the material and has provided diagrams showing the relation between q and r_n for different materials.

With regard to the q factor, two questions require further consideration. First, it is suggested that r_n does not constitute a complete representation of a notch. In addition, a comparison of experimental data with the above mentioned diagrams for different materials indicates that q is very sensitive to experimental errors and scatter of data. Based on data where statistical confidence limits on the fatigue strength have been estimated, the relation among q, r_n, and the material properties as suggested by Peterson requires further examination.

In this paper, the relation between K_f and K_t is considered from a new point of view based on the concept of "highly stressed volume" V, as reported in a previous work (**2**), which is summarized below together with results of the application of the concept to size and shape effects. The feasibility of this approach has also been contemplated by Peterson (**3**). The present paper considers the problem in terms of V_s and V_n, the highly stressed volume in the smooth and notched specimens, respectively.

THE CONCEPT OF HIGHLY STRESSED VOLUME

From an engineering point of view, measurements of fatigue strength in laboratory experiments are valid and useful to the extent that these values can be translated from small specimens generally used in this type of test to actual structural parts.

To translate values of fatigue strength from small laboratory specimens to structural members, the influence of the following variables, among others, must be considered: stress concentration due to notches, the variation of the fatigue strength with the volume (size effect), the shape of the actual element (which may differ from the shape of the fatigue specimen), and the possible difference in the method of application of the loads.

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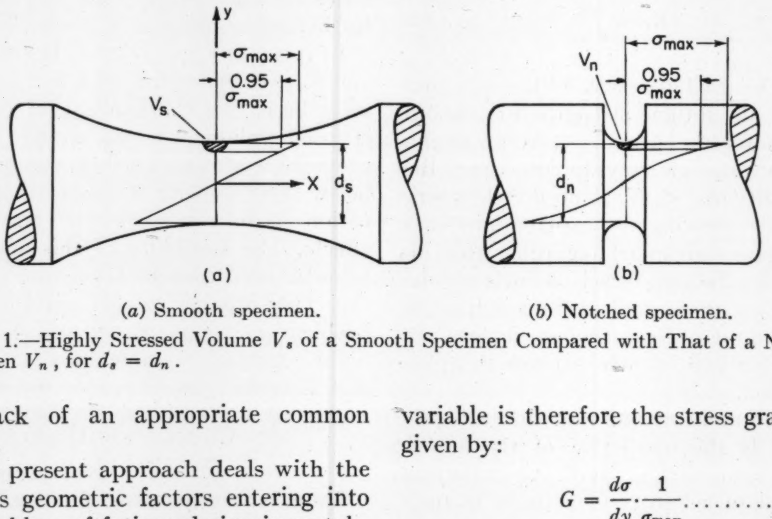
[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

Each one of these factors has received the careful attention of many investigators, each of whom dealt with the design problem from a particular point of view—the influence of the notch, the decrease of the fatigue strength with the diameter, the behavior of different shapes under repeated stresses, and so on. Unfortunately, the results have not always been in complete agreement and have never been compared because of the lack of an appropriate common factor.



(*a*) Smooth specimen.

(*b*) Notched specimen.

FIG. 1.—Highly Stressed Volume V_s of a Smooth Specimen Compared with That of a Notched Specimen V_n, for $d_s = d_n$.

The present approach deals with the various geometric factors entering into the problem of fatigue design in metals, namely, the notch, the size and shape effects, and the influence of different types of loading. The object is to find a simple expression with sufficient generality and relative accuracy to make it applicable in engineering design.

As a common factor or parameter, the influence of the highly stressed volume of material was investigated. Although a general procedure is outlined, the scope of this study is concerned primarily with the bending case, owing to the lack of appropriate data for other types of loading.

Stress Gradient:

The surface, where fatigue cracks are most frequently initiated, has a great influence in fatigue fracture. Hence, the condition of the surface deserves careful attention in any study directed toward a design point of view.

For a given metal, the surface could be defined knowing the following variables: (1) environment, (2) roughness, (3) residual stresses, and (4) stress gradient. It will be supposed that the first three factors are constant; the remaining variable is therefore the stress gradient, given by:

$$G = \frac{d\sigma}{dy} \cdot \frac{1}{\sigma_{max}} \qquad (4)$$

where y and σ_{max} are as defined in Fig. 1. Values of G for smooth specimens in bending are immediately obtained from Eq 4 as:

$$G = \frac{2}{d} \qquad (5)$$

It is possible to derive suitable expressions for other types of specimens from Neuber's formulas (4). For specimens with deep notches subjected to bending or twisting moments, the derivations are presented by Uzhik (5). Approximate

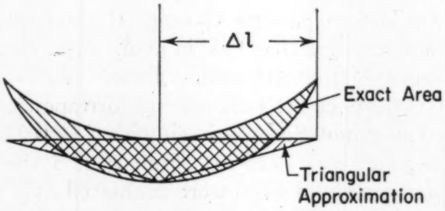


FIG. 2.—The Assumed Exact Area A and Its Triangular Approximation.

equations for computing G are given by Seely and Smith (6).

Statistical Approach:

From another point of view, fatigue failure is a statistical phenomenon. If failures are associated with flaws or defects in the material, the probability of finding a flaw is proportional to the volume of material. Therefore, the probability of failure increases with the volume of material. This logical argument is the basis for several statistical theories of fatigue (7,8).

Highly Stressed Volume of Material:

It is possible to combine the foregoing premises, thus demonstrating that, within a certain statistical margin, two specimens have the same probability of failure if both have the same highly stressed volume of material. By "highly stressed volume" is meant the thin layer of material subjected to a relatively high stress, for example, 95 per cent and more of $\sigma_{\max}$. For the approximation attained in this study, any region, or percentage, may be selected, provided a fairly linear stress gradient exists in that region. From the statistical point of view, the fatigue strength should no longer depend, among other factors, on the diameter itself, but on the highly stressed volume. In addition, it should be emphasized that it is possible to get the same amount of this volume in a small unnotched specimen as in a properly dimensioned larger specimen with a notch.

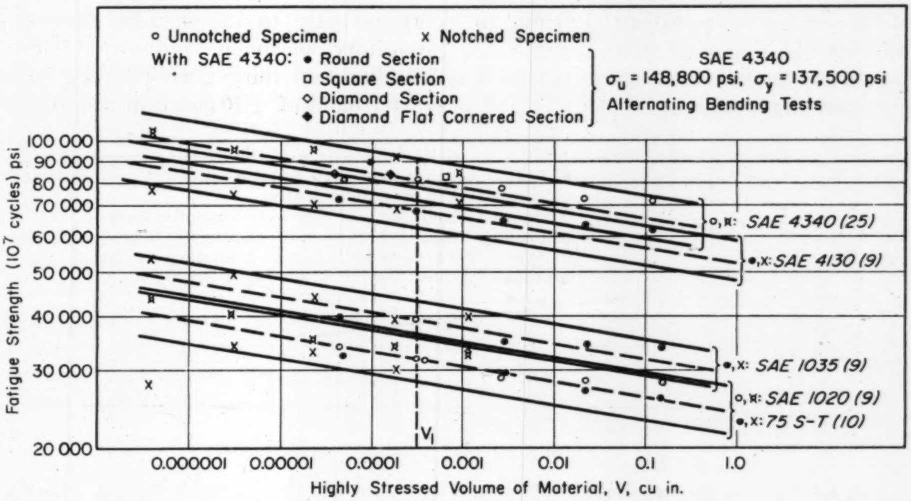


FIG. 3.—Relation Between Maximum Stress, $K_t\sigma_{\text{nom}}$, and Highly Stressed Volume of Material.

Size Effect:

The highly stressed volume hypothesis was applied first to size effect, that is, the decrease of the fatigue strength observed in metal specimens when the diameter is increased. Data from papers by Morkovin and Moore (9), and Moore and Dolan (10), were reexamined. The volume V was determined by taking into consideration the stress gradient and the following simplifying assumptions (see Figs. 1, 2, and 7):

1. The areas A will be considered triangular as shown in Fig. 2.
2. The distance d' between centroids of the areas A is approximately equal to the diameter d as illustrated in Fig. 7.
3. The volume V is subjected to a pure bending moment.
4. The factor K_t is not modified in magnitude for points on the surface between d and d_1 in Fig. 7.

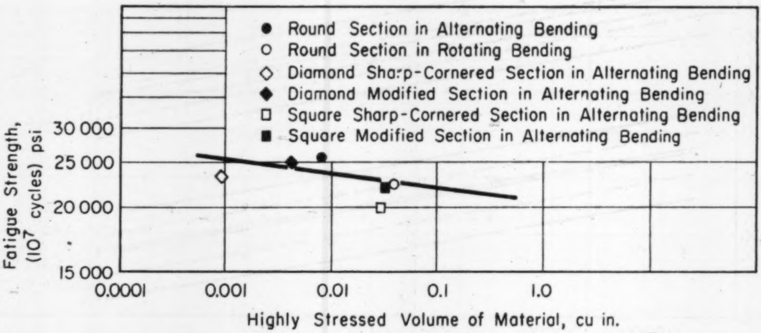


FIG. 4.—Relation Between Maximum Stress, $K_t\sigma_{\text{nom}}$, and Highly Stressed Volume of Material for 24 S-T Aluminum Alloy (13).

Hypotheses 3 and 4 depend for justification on the very small distance Δl involved in the problem. Determinations of the volume V are given in Appendices I and II.[3, 4] A noticeable decrease of the fatigue strength with the volume is illustrated in Fig. 3, where log V is plotted against log $\sigma_{\max}$. The relationship is fairly linear. The straight line can be represented by the equation:

$$\sigma_{\max} = BV^{-a} \quad \ldots\ldots\ldots\ldots (6)$$

In order to make this procedure applicable in engineering problems, this line was drawn passing through the experimental point that has, in every case, the same highly stressed volume: V_1 = 0.000,349 cu in. This volume corresponds to an unnotched specimen with d = 0.25 in., and r = 1.25 in. From Fig. 3 the parameters in Eq 6 were evaluated as:

$$B = 0.763\,\sigma_{\max 1}$$

where $\sigma_{\max 1}$ is the maximum stress for V_1, corresponding to the 0.25-in. diameter laboratory specimen, and a = 0.034. This line and those corresponding to a scatter band of ±10 per cent are drawn in Fig. 3. All the data points except two lie within this scatter band. The application of the concept of the highly stressed volume to the size effect was previously discussed by Fowler (11) and Stulen (12). They showed that size effect depends not only on the volume of the highly stressed material, but also the scatter (as measured by the standard deviation) of the fatigue strength of the reference specimen.

Shape Effect and Type of Loading:

According to the procedure followed to determine the highly stressed volume, application of the preceding analysis is not restricted to specimens having the same cross-sectional shapes or to a particular type of loading. Therefore, the technique was extended by considering data from Corten and Dolan (13) and Dolan, McClow, and Craig (14). Figure 4 shows the behavior of 24S-T aluminum alloy specimens under flexural and rotating - bending tests (13). Since in this case there are no data for specimens with the "index volume," that is, a volume $V_1 = 0.000{,}349$ cu in., it is not possible to apply Eq 6. Nevertheless, it is shown that the specimens follow the general trend. From Fig. 4 it is clear that the sharp-cornered sections exhibit a very definite reduction of strength, in addition to the effect of the highly stressed volume. In Fig. 3 data for an SAE 4340 steel are shown corresponding to four different shapes of cross-section under alternated bending, according to Dolan, McClow, and Craig (14). These data lie within the same scatter band as determined for the other SAE 4340 steel (10). No correction was made for the difference in the static properties.

THE RELATION BETWEEN THE THEORETICAL STRESS CONCENTRATION FACTOR K_t AND THE FATIGUE NOTCH FACTOR K_f

The relation between K_t and K_f will be considered in terms of the highly stressed volume concept. Equation 6 may be substituted into Eq 2 to give a new relation for K_f :

$$K_f = \frac{\sigma_{fs}}{\sigma_{fn}} = \frac{BV_s^{-a}}{\dfrac{BV_n^{-a}}{K_t}} = K_t\left(\frac{V_s}{V_n}\right)^{-a}$$

Finally:

$$\frac{K_f}{K_t} = \left(\frac{V_n}{V_s}\right)^{0.034} \ldots\ldots\ldots\ldots (7)$$

Considering both smooth and notched specimens of the same diameter, it is evident from Fig. 1 that V_n will always be smaller than V_s ; consequently, K_f will always be smaller than K_t. Thus, from Eq 7 is deduced the well-known experimental inequality, $K_f < K_t$.

Quantitative evaluation of K_f based on Eq 7 will now be undertaken for members subjected to rotating bending and axial repeated loading.

Shallow Notched Specimens in Rotating Bending:

For a specimen of diameter d and profile radius r, the general expression for V in rotating bending is given (see Appendix I[3]) by:

$$V \cong 0.02\,\frac{\sqrt{rd}\,d}{G} \ldots\ldots\ldots\ldots (8)$$

For smooth specimens, $G = \dfrac{2}{d_s}$, and Eq 8 becomes

$$V_s \cong 0.01\,\sqrt{r_s d_s^5} \ldots\ldots\ldots (9)$$

And for shallow notched specimens, $G = \dfrac{2.5}{r_n}$, and Eq 8 is

$$V_n \cong 0.008\,\sqrt{r_n^3 \cdot d_n^3} \ldots\ldots\ldots (10)$$

The ratio K_f/K_t from Eq 7 may be written, using Eqs 9 and 10, as:

$$\frac{K_f}{K_t} = \left(0.8\,\sqrt{\frac{r_n^3 d_n^3}{r_s d_s^5}}\right)^{0.034}$$

and, for $d_n = d_s = d$,

$$\frac{K_f}{K_t} = \left(0.8\,\sqrt{\frac{r_n^3}{r_s d^2}}\right)^{0.034}$$

$$= (0.8)^{0.034}\left(\frac{r_s^{0.051}}{r_s^{0.017} d^{0.034}}\right) \ldots (11)$$

Thus, the ratio K_f/K_t depends solely upon the geometry of the critical section of both smooth and notched specimens, and not upon the material. Further consideration of the influence of the material is given later in the Discussion.

The influence of each of the geometric

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[3] See p. 745.

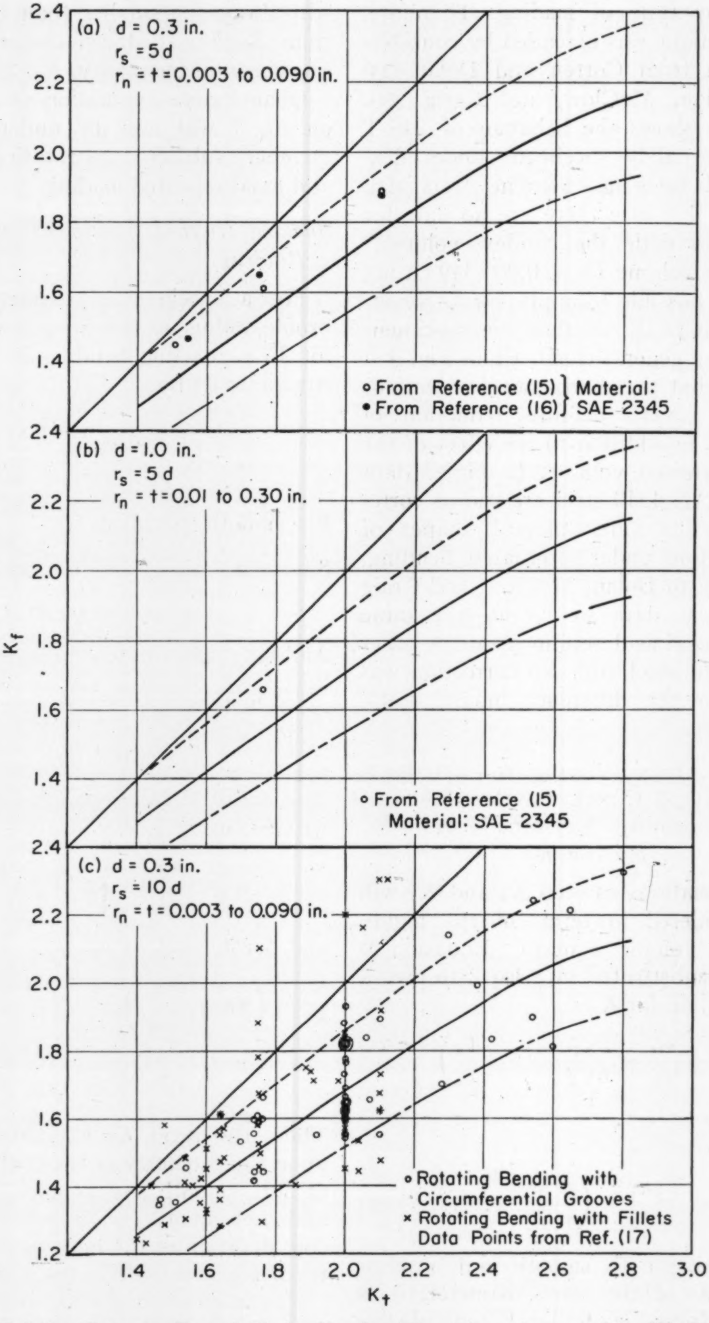

FIG. 5.—Relation Between K_t and K_f for Varying Dimensions d, r_s, and r_n (K_t Determined According to Peterson (1, p. 47)).

characteristics, r_n, d, and r_s, can be estimated from the following expression:

$$\frac{d(K_f/K_t)}{(K_f/K_t)} = \pm \frac{\frac{\partial(K_f/K_t)}{\partial r_n} dr_n}{K_f/K_t} \pm \frac{\frac{\partial(K_f/K_t)}{\partial r_s} dr_s}{K_f/K_t} \pm \frac{\frac{\partial(K_f/K_t)}{\partial d} dd}{K_f/K_t}$$

Replacing the derivatives by their values, and simplifying:

$$\frac{d(K_f/K_t)}{(K_f/K_t)} = \pm 0.051 \frac{dr_n}{r_n} \pm 0.017 \frac{dr_s}{r_s} \pm 0.034 \frac{dd}{d}$$

Therefore, a unit variation of r_n has three times the effect of r_s, and 1.5 times that of d, on the variation of K_f/K_t.

For the purpose of discussion, it is convenient to consider specific illustrations.

Example I.—Determine the relation between K_f and K_t for specimens with the following dimensions $d = 0.3$ in. and $r_s = 5\,d$.

Equation 11 becomes

$$Kf = K_t(2.177\sqrt{r_n^3})^{0.034} = 1.0268K_t(r_n)^{0.051} \quad (12)$$

In Fig. 5(*a*) the relation between K_f and K_t given by Eq 12 is shown. The values of K_t (determined from Peterson (**1**)) correspond to a range of notch radii, r_n, from 0.003 to 0.090 in. In addition, a scatter band enclosing a region of ±10 per cent on either side of the curve representing Eq 12 is shown.

Experimental data (**15,16**) are also shown in Fig. 5(*a*). The data lie above the values given by Eq 12 but well within the scatter band. The significance of the difference between the data and the curve representing Eq 12 will be considered in detail later.

Example II.—The relation between K_f and K_t for specimens with dimensions $d = 1$ in. and $r_s = 5\,d$ is given by Eq 11 as

$$K_f = K_t(0.358\sqrt{r_n^3})^{0.034} = 0.9656K_t(r_n)^{0.051} \quad (13)$$

The relation between K_f and K_t given by Eq 13 is shown in Fig. 5(*b*). Experimental data extracted from Moore and Jordan (**15**) lie above the curve but within the ±10 per cent scatter band.

Example III.—The relation between K_t and K_f for specimens with dimensions $d = 0.3$ in., and $r_s = 10\,d$ is given by Eq 11 as

$$K_f = K_t(1.5396\sqrt{r_n^3})^{0.034} = 1.0148K_t(r_n)^{0.051} \quad (14)$$

The relation between K_f and K_t given by Eq 14 is shown in Fig. 5(*c*).

As noted previously, the influence of the notch radius r_n on the ratio K_f/K_t is largest, followed in decreasing importance by d and r_s. The range of values of d and r_s encountered in most investigations of notch sensitivity has been limited frequently to a range no larger than that considered in the above examples. From Eqs 12, 13, and 14, the largest difference between values of K_f was less than 6 per cent and this difference occurred when the value of d was changed from $d = 0.3$ in. to $d = 1.0$ in. It is possible to determine the variation of the numerical coefficient of Eqs 12, 13, and 14 from the expression

$$C = \frac{0.8^{0.034}}{r_s^{0.017} d^{0.034}}$$

for extreme values of d and r_s that might be used in fatigue specimens. For example, for a small specimen, $d = 0.125$ in. and $r_s = 3d = 0.375$ in., the value of C is 1.1083. For a large specimen, $d = 1$ in. and $r_s = 10d = 10$ in., C is 0.9544.

Thus, a wide variation of the geometric characteristics of smooth fatigue specimens produces only approximately 15 per cent change in C. Naturally, the difference would be larger if an actual structural or machine member were considered. From this, it may be concluded that most of the data in the literature on notch sensitivity will exhibit such a small influence due to changes of d and r_s that the difference is not measurable except possibly when many specimens are tested and analyzed statistically. Hence, Fig. 5(*c*) has been taken as the basic curve for comparison with data for a large number of similar types of specimens. The experimental values have been extracted from Kuhn and Hardrath (**17**) for a range of values of K_t between 1.4 and 2.8. A total of 112 sets of data summarizing values of K_f and K_t have been included.

TABLE I.—COMPUTED VALUES COMPARED WITH EXPERIMENTAL VALUES OF K_f.

K_t	K_f, Computed	K_f, Experimental		
		For 24S-T3 Aluminun Alloy	For 75S-T6 Aluminum Alloy	For SAE 4130 Steel
2.......	1.8	1.8	1.9	1.9
2.......	1.6	1.8	1.9	1.7
2.......	1.6	1.8	1.9	1.7
4.......	3.0	3.1	4.0	3.4
4.......	2.8	2.4	3.2	2.8
5.......	3.6	3.4	5.0	4.7

Of the 112 registered points in Fig. 5(*c*), 87 (78 per cent) lie within the ±10 per cent scatter band.

Plates Loaded Repeatedly in Axial Tension-Compression:

The volumes V_s and V_n required to evaluate Eq 7 are given (see Appendix II[4]) by:

$$V_s = 0.46\sqrt{r_s b_s} t b_s$$

for unnotched specimens. Similarly,

$$V_n = 0.018\sqrt{r_n b_n} t r_n$$

for shallow notched specimens, and

$$V_n = 0.024\sqrt{r_n b_n} t r_n$$

for deep notched specimens.

These equations were used to determine the values of K_f for plates subjected to axial loads. The computed values are compared with experimental values extracted from Grover, Bishop, and Jackson (**18, 19**), in Table I. A ±10 per cent scatter band includes 56 per cent of the 18 sets of experimental data.

DISCUSSION

Basis for Selection of the ±10 Per Cent Scatter Band:

Two characteristics of fatigue data that affect the accuracy of measurement of notch sensitivity are the large scatter of data and the possible large influence of several "incidental" variables.

Several of the factors that produce systematic errors will be reviewed briefly, followed by a discussion of the statistical variability of data.

Method of Preparation of Test Specimens.—The fatigue strengths of specimens made from the same material vary significantly if the specimens are machined and finished in different machine shops, or by different operators in the same machine shop. The irregular behavior is caused by variations in residual stresses, surface cold working, and surface roughness introduced by the different techniques employed in machining and polishing the specimens.

Gohn and Fox have reported (**20**), for example, a difference of 25 per cent in the fatigue strength of hard-drawn grade A phosphor-bronze specimens in rotating-bending tests machined by two different machine shops, both in accordance with the methods given in the ASTM Manual on Fatigue Testing (**21**).

Even when all specimens are prepared by the same operator, the difference in root radii between unnotched and notched specimens usually makes it impossible to employ similar machining and polishing techniques on the two types of specimens. These factors may introduce systematic differences and an inherent lack of reproducibility between otherwise comparable experimental determinations of notch sensitivity.

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[4] See p. 746.

Influence of Normally Uncontrolled Environmental Conditions During Testing.—Most of the fatigue tests performed at room temperature are without accurate control of such periodically varying environmental conditions as temperature and water content of the air in the laboratory. Nevertheless, these variables may have a small but consistent influence on the life of the specimens.

TABLE II.—RESULTS OF CONTROLLED FATIGUE TESTS.

Material	σ_f, psi	±0.10 σ_f, psi	±1 Standard Deviation, psi	±2 Standard Deviations, psi
SAE 1050 steel.....	40 800	4100	1000	2 000
Annealed Armco iron...........	29 800	3000	2300	4 600
Quenched and spheroidized SAE 4340 steel.......	62 500	6300	2700	5 400
Quenched and tempered SAE 4340 steel...........	97 600	9800	7200	14 400

For instance, Liu and Corten have found (22) that the water content of the air influences the life of uncoated aluminum alloy specimens under repeated stresses: an increase of water content from 12 to 120 grains per lb of dry air decreased the fatigue life by a factor of approximately 2.

Statistical Nature of Fatigue.—The most important source of random scatter in the measurement of K_f is the statistical nature of the fatigue phenomenon itself. Heterogeneities in the material are the sources for the origin of fatigue cracks, and hence of fatigue fracture. Individual specimens of a single statistical population exhibit a large range of scatter in life and a corresponding scatter in fatigue strength.

In the experimental determinations of K_f summarized in Fig. 5(*c*), it was common practice to base the value of K_f on the results of several specimens tested in the region of the fatigue limit (or fatigue strength at 10^7 cycles). Each investigation employed a larger number of specimens, but the value of K_f was determined by the few specimens that were tested at stresses very close to the fatigue strength. Thus, it is appropriate to inquire as to how much scatter may be present in the above mentioned results by examining the statistical variability, that is, the standard deviation, that must be associated with the fatigue strength.

Carefully planned and controlled tests (23) in which all but the statistical sources of scatter were minimized gave the results shown in Table II. Table II indicates that a ±10 per cent scatter band covers a range slightly larger than 1 standard deviation for two sets of data, slightly larger than 2 standard deviations for a third set of data, and about 4 standard deviations for the fourth set of data.

In view of the fact that the quantity K_f is the ratio of two values of fatigue strength, it is appropriate to inquire how the standard deviation for K_f is related to the standard deviation of either value of fatigue strength. For a quotient such as

$$K_f = \frac{\sigma_{fs}}{\sigma_{fn}} \ldots\ldots\ldots\ldots\ldots(2)$$

the standard deviation S_{K_f} is given by Volk (26):

$$S_{K_f}^2 = K_f^2 \frac{S_{\sigma_{fs}}^2}{\bar{\sigma}_{fs}^2} + \bar{K}_f^2 \frac{S_{\sigma_{fn}}^2}{\bar{\sigma}_{fn}^2} \ldots\ldots(15)$$

where $\bar{K}_f$, $\bar{\sigma}_{fs}$, and $\bar{\sigma}_{fn}$ are, respectively, the mean values of K_f, σ_{fs}, and σ_{fn}. Dividing both sides of Eq 15 by $\bar{K}_f^2$ and substituting $(\bar{K}_f\bar{\sigma}_{fn})$ for $\bar{\sigma}_{fs}$ gives

$$\frac{S_{K_f}^2}{\bar{K}_f^2} = \frac{S_{\sigma_{fs}}^2}{\bar{K}_f^2\bar{\sigma}_{fn}^2} + \frac{S_{\sigma_{fn}}^2}{\bar{\sigma}_{fn}^2}$$

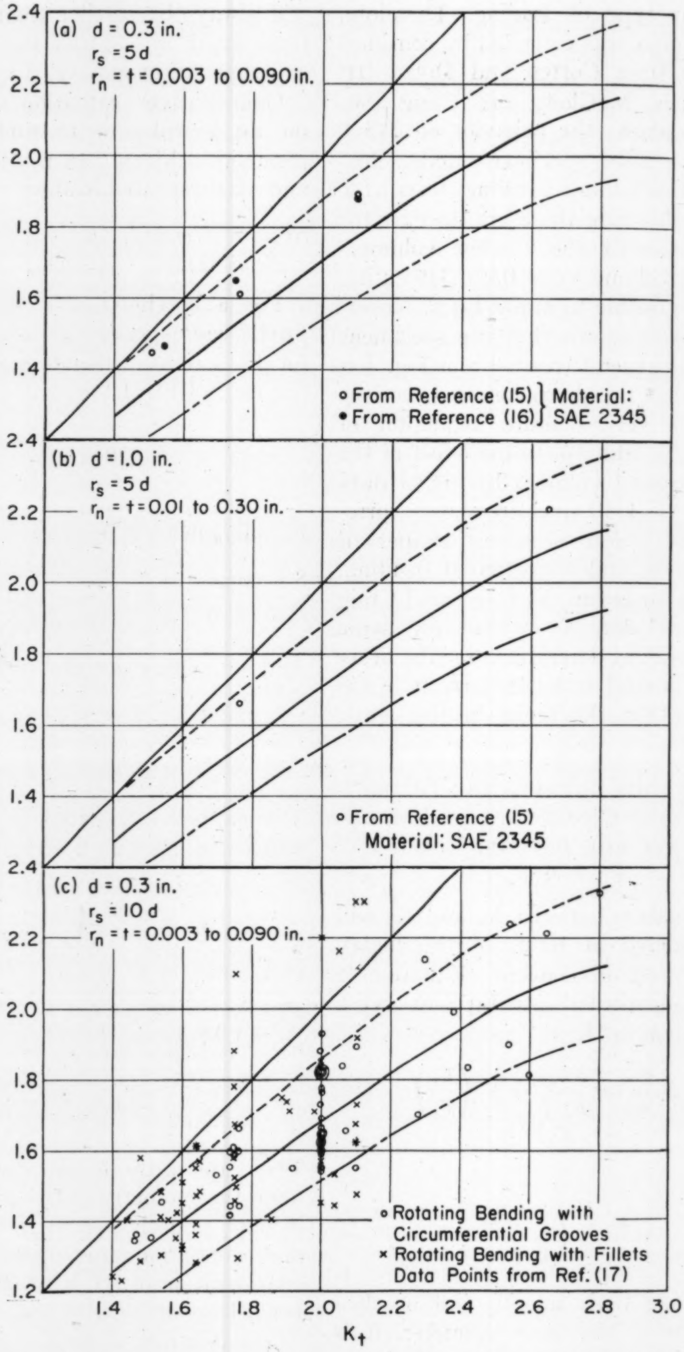

FIG. 5.—Relation Between K_t and K_f for Varying Dimensions d, r_s, and r_n (K_t Determined According to Peterson (1, p. 47)).

characteristics, r_n, d, and r_s, can be estimated from the following expression:

$$\frac{d(K_f/K_t)}{(K_f/K_t)} = \pm \frac{\frac{\partial(K_f/K_t)}{\partial r_n} dr_n}{K_f/K_t} \pm \frac{\frac{\partial(K_f/K_t)}{\partial r_s} dr_s}{K_f/K_t} \pm \frac{\frac{\partial(K_f/K_t)}{\partial d} dd}{K_f/K_t}$$

Replacing the derivatives by their values, and simplifying:

$$\frac{d(K_f/K_t)}{(K_f/K_t)} = \pm 0.051 \frac{dr_n}{r_n} \pm 0.017 \frac{dr_s}{r_s} \pm 0.034 \frac{dd}{d}$$

Therefore, a unit variation of r_n has three times the effect of r_s, and 1.5 times that of d, on the variation of K_f/K_t.

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In Fig. 5(a) the relation between K_f and K_t given by Eq 12 is shown. The values of K_t (determined from Peterson (**1**)) correspond to a range of notch radii, r_n, from 0.003 to 0.090 in. In addition, a scatter band enclosing a region of ±10 per cent on either side of the curve representing Eq 12 is shown.

Experimental data (**15,16**) are also shown in Fig. 5(a). The data lie above the values given by Eq 12 but well within the scatter band. The significance of the difference between the data and the curve representing Eq 12 will be considered in detail later.

Example II.—The relation between K_f and K_t for specimens with dimensions $d = 1$ in. and $r_s = 5\,d$ is given by Eq 11 as

$$K_f = K_t(0.358\sqrt{r_n^3})^{0.034} = 0.9656K_t(r_n)^{0.051} \quad (13)$$

The relation between K_f and K_t given by Eq 13 is shown in Fig. 5(b). Experimental data extracted from Moore and Jordan (**15**) lie above the curve but within the ±10 per cent scatter band.

Example III.—The relation between K_t and K_f for specimens with dimensions $d = 0.3$ in., and $r_s = 10\,d$ is given by Eq 11 as

$$K_f = K_t(1.5396\sqrt{r_n^3})^{0.034} = 1.0148K_t(r_n)^{0.051} \quad (14)$$

The relation between K_f and K_t given by Eq 14 is shown in Fig. 5(c).

As noted previously, the influence of the notch radius r_n on the ratio K_f/K_t is largest, followed in decreasing importance by d and r_s. The range of values of d and r_s encountered in most investigations of notch sensitivity has been limited frequently to a range no larger than that considered in the above examples. From Eqs 12, 13, and 14, the largest difference between values of K_f was less than 6 per cent and this difference occurred when the value of d was changed from $d = 0.3$ in. to $d = 1.0$ in. It is possible to determine the variation of the numerical coefficient of Eqs 12, 13, and 14 from the expression

$$C = \frac{0.8^{0.034}}{r_s^{0.017} d^{0.034}}$$

for extreme values of d and r_s that might be used in fatigue specimens. For example, for a small specimen, $d = 0.125$ in. and $r_s = 3d = 0.375$ in., the value of C is 1.1083. For a large specimen, $d = 1$ in. and $r_s = 10d = 10$ in., C is 0.9544.

Thus, a wide variation of the geometric characteristics of smooth fatigue specimens produces only approximately 15 per cent change in C. Naturally, the difference would be larger if an actual structural or machine member were considered. From this, it may be concluded that most of the data in the literature on notch sensitivity will exhibit such a small influence due to changes of d and r_s that the difference is not measurable except possibly when many specimens are tested and analyzed statistically. Hence, Fig. 5(*c*) has been taken as the basic curve for comparison with data for a large number of similar types of specimens. The experimental values have been extracted from Kuhn and Hardrath (**17**) for a range of values of K_t between 1.4 and 2.8. A total of 112 sets of data summarizing values of K_f and K_t have been included.

TABLE I.—COMPUTED VALUES COMPARED WITH EXPERIMENTAL VALUES OF K_f.

K_t	K_f, Computed	K_f, Experimental		
		For 24S-T3 Aluminun Alloy	For 75S-T6 Aluminum Alloy	For SAE 4130 Steel
2	1.8	1.8	1.9	1.9
2	1.6	1.8	1.9	1.7
2	1.6	1.8	1.9	1.7
4	3.0	3.1	4.0	3.4
4	2.8	2.4	3.2	2.8
5	3.6	3.4	5.0	4.7

Of the 112 registered points in Fig. 5(*c*), 87 (78 per cent) lie within the ±10 per cent scatter band.

Plates Loaded Repeatedly in Axial Tension-Compression:

The volumes V_s and V_n required to evaluate Eq 7 are given (see Appendix II[4]) by:

$$V_s = 0.46\sqrt{r_s b_s}\,tb_s$$

for unnotched specimens. Similarly,

$$V_n = 0.018\sqrt{r_n b_n}\,tr_n$$

for shallow notched specimens, and

$$V_n = 0.024\sqrt{r_n b_n}\,tr_n$$

for deep notched specimens.

These equations were used to determine the values of K_f for plates subjected to axial loads. The computed values are compared with experimental values extracted from Grover, Bishop, and Jackson (**18, 19**), in Table I. A ±10 per cent scatter band includes 56 per cent of the 18 sets of experimental data.

Discussion

Basis for Selection of the ±10 Per Cent Scatter Band:

Two characteristics of fatigue data that affect the accuracy of measurement of notch sensitivity are the large scatter of data and the possible large influence of several "incidental" variables.

Several of the factors that produce systematic errors will be reviewed briefly, followed by a discussion of the statistical variability of data.

Method of Preparation of Test Specimens.—The fatigue strengths of specimens made from the same material vary significantly if the specimens are machined and finished in different machine shops, or by different operators in the same machine shop. The irregular behavior is caused by variations in residual stresses, surface cold working, and surface roughness introduced by the different techniques employed in machining and polishing the specimens.

Gohn and Fox have reported (**20**), for example, a difference of 25 per cent in the fatigue strength of hard-drawn grade A phosphor-bronze specimens in rotating-bending tests machined by two different machine shops, both in accordance with the methods given in the ASTM Manual on Fatigue Testing (**21**).

Even when all specimens are prepared by the same operator, the difference in root radii between unnotched and notched specimens usually makes it impossible to employ similar machining and polishing techniques on the two types of specimens. These factors may introduce systematic differences and an inherent lack of reproducibility between otherwise comparable experimental determinations of notch sensitivity.

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[4] See p. 746.

Influence of Normally Uncontrolled Environmental Conditions During Testing.—Most of the fatigue tests performed at room temperature are without accurate control of such periodically varying environmental conditions as temperature and water content of the air in the laboratory. Nevertheless, these variables may have a small but consistent influence on the life of the specimens.

TABLE II.—RESULTS OF CONTROLLED FATIGUE TESTS.

Material	σ_f, psi	± 0.10 σ_f, psi	± 1 Standard Deviation, psi	± 2 Standard Deviations, psi
SAE 1050 steel.....	40 800	4100	1000	2 000
Annealed Armco iron............	29 800	3000	2300	4 600
Quenched and spheroidized SAE 4340 steel.......	62 500	6300	2700	5 400
Quenched and tempered SAE 4340 steel............	97 600	9800	7200	14 400

For instance, Liu and Corten have found (22) that the water content of the air influences the life of uncoated aluminum alloy specimens under repeated stresses: an increase of water content from 12 to 120 grains per lb of dry air decreased the fatigue life by a factor of approximately 2.

Statistical Nature of Fatigue.—The most important source of random scatter in the measurement of K_f is the statistical nature of the fatigue phenomenon itself. Heterogeneities in the material are the sources for the origin of fatigue cracks, and hence of fatigue fracture. Individual specimens of a single statistical population exhibit a large range of scatter in life and a corresponding scatter in fatigue strength.

In the experimental determinations of K_f summarized in Fig. 5(*c*), it was common practice to base the value of K_f on the results of several specimens tested in the region of the fatigue limit (or fatigue strength at 10^7 cycles). Each investigation employed a larger number of specimens, but the value of K_f was determined by the few specimens that were tested at stresses very close to the fatigue strength. Thus, it is appropriate to inquire as to how much scatter may be present in the above mentioned results by examining the statistical variability, that is, the standard deviation, that must be associated with the fatigue strength.

Carefully planned and controlled tests (23) in which all but the statistical sources of scatter were minimized gave the results shown in Table II. Table II indicates that a ±10 per cent scatter band covers a range slightly larger than 1 standard deviation for two sets of data, slightly larger than 2 standard deviations for a third set of data, and about 4 standard deviations for the fourth set of data.

In view of the fact that the quantity K_f is the ratio of two values of fatigue strength, it is appropriate to inquire how the standard deviation for K_f is related to the standard deviation of either value of fatigue strength. For a quotient such as

$$K_f = \frac{\sigma_{fs}}{\sigma_{fn}} \quad \ldots\ldots\ldots\ldots (2)$$

the standard deviation S_{K_f} is given by Volk (26):

$$S_{K_f}^2 = K_f^2 \frac{S_{\sigma_{fs}}^2}{\bar{\sigma}_{fs}^2} + \bar{K}_f^2 \frac{S_{\sigma_{fn}}^2}{\bar{\sigma}_{fn}^2} \quad \ldots\ldots (15)$$

where $\bar{K}_f$, $\bar{\sigma}_{fs}$, and $\bar{\sigma}_{fn}$ are, respectively, the mean values of K_f, σ_{fs}, and σ_{fn}. Dividing both sides of Eq 15 by $\bar{K}_f^2$ and substituting $(\bar{K}_f\bar{\sigma}_{fn})$ for $\bar{\sigma}_{fs}$ gives

$$\frac{S_{K_f}^2}{\bar{K}_f^2} = \frac{S_{\sigma_{fs}}^2}{\bar{K}_f^2\bar{\sigma}_{fn}^2} + \frac{S_{\sigma_{fn}}^2}{\bar{\sigma}_{fn}^2}$$

For convenience, let $S_{\sigma_{fs}} = \alpha S_{\sigma_{fn}}$, where frequently $\alpha > 1$, since notched specimens exhibit less scatter than unnotched specimens. Then

$$\frac{S_{K_f}}{\bar{K}_f} = \sqrt{\left(\frac{\alpha}{\bar{K}_f}\right)^2 + 1}\left(\frac{S_{\sigma_{fn}}}{\bar{\sigma}_{fn}}\right) \ldots (16)$$

or

$$\frac{S_{K_f}}{\bar{K}_f} = \sqrt{1 + \left(\frac{\bar{K}_f}{\alpha}\right)^2}\left(\frac{S_{\sigma_{fs}}}{\bar{\sigma}_{fs}}\right) \ldots (17)$$

and so on, do not coincide to give a rather unusual lack of dispersion.

From the data in Fig. 5(*c*), where 78 per cent of 112 sets of data were included within the scatter band, it may be estimated that the ±10 per cent band corresponds approximately to ±1.2 standard deviations.

A more accurate analysis of scatter is desirable; however, at the present time adequate statistical data are scarce.

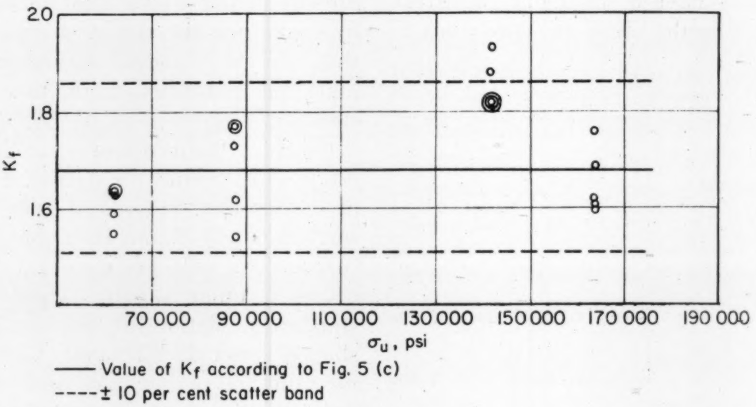

FIG. 6.—K_f versus σ_u for SAE 1020, SAE 1035, SAE X4130, and SAE 4340 Steels for $K_t = 2$ (According to Data from References (**24**) and (**25**)).

From Eq 16 or 17 it is obvious that the ratio $S_{K_f}/\bar{K}_f$ is larger than either of the ratios $S_{\sigma_{fs}}/\bar{\sigma}_{fs}$ or $S_{\sigma_{fn}}/\bar{\sigma}_{fn}$. This is necessary, since the value of the term under the square root in Eq 16 or 17 is necessarily larger than one. Thus, in terms of a scatter band, the width of the scatter band of K_f expressed as a percentage of K_f is larger than the scatter band associated with either $\bar{\sigma}_{fs}$ or $\bar{\sigma}_{fn}$.

Once having accumulated the above-mentioned sources of dispersion, it may be concluded that the ±10 per cent scatter band gives a conservative estimate of the scatter that must be expected in determinations of K_f whenever the occurrence of special characteristics of the material, geometry of the specimen,

Scope and Limitations of the Present Approach:

It is the author's belief that factors other than the geometric characteristics included in the calculation of the volumes V have a small influence on the relation between K_f and K_t. Hardness of the material, heterogeneity of the crystalline structure, and localized inelastic deformation probably influence this relation.

Considering the amount of scatter that must be expected in the determination of K_f, however, it appears impossible to evaluate these factors from available data. For example, the influence of the tensile strength on the value of K_f is shown in Fig. 6. While it appears that the value of K_f tends to increase ($K_t = 2.0$ for all data) with tensile strength, the trend is not consistent. Note particularly that 18 of the 20 points are included within a ±10 per cent band, a band that is independent of tensile strength.

For the purpose of estimating an appropriate value of K_f for use in design, it is suggested that the value of K_t be modified by the "highly stressed volume" concept as given in Eq 7. Based on the data of Fig. 5(*c*), it may be anticipated that in four out of five practical design cases the value of K_f will be within ±10 per cent of an experimentally measured value.

CONCLUSIONS

The results of this study appear to justify the following conclusions:

1. The fatigue strength of metals decreases when the stressed volume of material at a surface layer increases. A highly stressed region—for example, a volume, V, subjected to 95 per cent more of $\sigma_{\max}$—may be used in computations for design purposes.

2. A plot of the logarithm of volume V against the logarithm of the maximum stress attained by a specimen gave a linear relationship with a fairly constant slope for all the materials reviewed. The equation of this line may be defined for a given material knowing only one value of fatigue strength: the maximum stress for $V_1 = 0.000,349$ cu in. which corresponds to a smooth specimen with $d = 0.25$ in. and $r = 1.25$ in. in a rotating-bending test.

By comparing this equation with existing data for both smooth specimens with different shapes and notched specimens in rotating and alternating bending tests, a ±10 per cent scatter band was found to include nearly all the data.

3. It is possible to infer the inequality $K_f < K_t$ from the "highly stressed volume" approach.

4. It is shown that K_f depends upon K_t, the root radii of the notched and smooth specimens, and the diameter d; however, the influence of K_t and the notch radius are of greatest significance.

5. The relation between K_f and K_t for rotating-bending specimens is given by by Eq 11 and shown in Fig. 5(*c*). Comparison with experimental data indicates that a ±10 per cent scatter band includes 78 per cent of the data. Based on these results, this relation is recommended for use in design.

6. Factors other than K_t and the highly stressed volume undoubtedly influence the value of K_f; however, based on available data, these factors are lost in the scatter present in the data.

7. Further studies employing statistical analysis on members of complex shape are desirable in order to corroborate more fully the concepts and design procedure proposed in this paper.

Acknowledgment:

This study was conducted in the Department of Theoretical and Applied Mechanics at the University of Illinois. A fellowship of the National Council of Scientific and Technical Research of the Argentine Republic made this work possible. Grateful acknowledgment is due H. T. Corten for his continued interest, encouragement, and valuable advice. Special acknowledgment is also due T. J. Dolan, Head of the Department of Theoretical and Applied Mechanics, for the facilities made available during the study.

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APPENDIX I

DETERMINATION OF HIGHLY STRESSED VOLUME, *V*, FOR ROUND-CROSS-SECTION SPECIMENS LOADED IN ROTATING BENDING

For the specimen shown in Fig. 7, the highly stressed volume, that is, material subjected to 95 per cent and more of $\sigma_{\max}$, can be taken, according to the simplified hypothesis given in the text, as

$$V = \pi\, d' A \ldots\ldots\ldots\ldots (18)$$

but,

$d' \cong d$ (that is, centroidal distance = diameter)

and

$$A \cong 2\,\frac{\Delta l h}{2} = \Delta l h$$

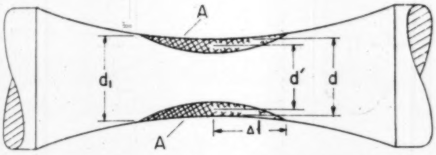

Fig. 7.—Diagram of the Areas *A* Subjected to 95 Per Cent and More of the Maximum Stress.

where *A* is the area formed by the intersection of the volume *V* with any plane containing the centerline of the specimen. The area *A* is computed by approximating it by a triangle of base $2\Delta l$ and height *h*.

Then in Eq 18:

$$V \cong \pi\, d \Delta l h \ldots\ldots\ldots\ldots (19)$$

The height *h* may be determined as follows: By definition

$$G = \frac{\sigma_{\max} - \sigma_{\min}}{h\sigma_{\max}}$$

In Fig. 8, $\sigma_{\min} = 0.95\,\sigma_{\max}$. Then

$$h = 0.05\,\frac{1}{G} \ldots\ldots\ldots\ldots (20)$$

The length Δl shown in Fig. 8 may be estimated from

$$\Delta l^2 = r^2 - \left(r - \frac{d_1 - d}{2}\right)^2 = r(d_1 - d) \ldots (21)$$

where the term $\left(\frac{d_1 - d}{2}\right)^2$ was neglected in this approximation due to its comparative smallness.

With regard to d_1 in Eq 21, it will be assumed that the bending moment *M* remains approximately constant over the region *d* to d_1. Therefore

$$M = \sigma W = \sigma_1 W_1$$

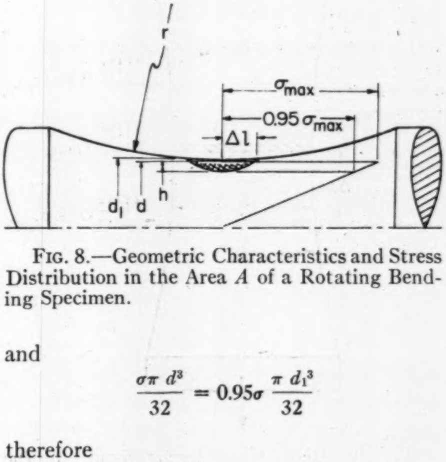

Fig. 8.—Geometric Characteristics and Stress Distribution in the Area *A* of a Rotating Bending Specimen.

and

$$\frac{\sigma\pi\, d^3}{32} = 0.95\sigma\,\frac{\pi\, d_1^3}{32}$$

therefore

$$d_1 = 1.017\, d$$

Then, replacing d_1 in Eq 21:

$$\Delta l \cong 0.13\sqrt{r\, d} \ldots\ldots\ldots\ldots (22)$$

Introducing Eqs 20 and 21 in Eq 19 gives

$$V \cong 0.02\,\frac{\sqrt{r\, d}}{G}\, d \ldots\ldots\ldots\ldots (23)$$

This expression is valid for unnotched specimens and notched specimens provided K_t remains approximately constant over the small distance, *d* to d_1.

APPENDIX II

DETERMINATION OF VOLUME V FOR PLATES SUBJECTED TO AN AXIAL LOAD

Consider the case of smooth specimens as shown in Fig. 9. The volume V_s is given by:

$$V_s = 2F\Delta l \quad \text{.............(24)}$$

where

$$F \cong tb_s \quad \text{.............(24a)}$$

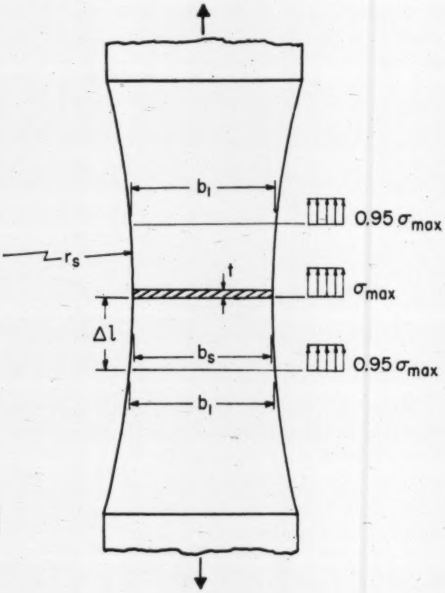

FIG. 9.—Geometric Characteristics of the Highly Stressed Volume of a Plate Specimen Under Axial Load.

and

$$\Delta l \cong \sqrt{r_s(b_1 - b_s)} \quad \text{........(24b)}$$

But

$$P = \sigma A = \sigma_1 A_1$$

Therefore

$$\sigma_{\max} b_s t = 0.95\sigma_{\max} b_1 t$$

and

$$b_1 = \frac{1}{0.95} b_s$$

Replacing b_1 in Eq 24*b* with this value gives

$$\Delta l \cong \frac{1}{\sqrt{19}} \sqrt{r_s b_s} \quad \text{.........(25)}$$

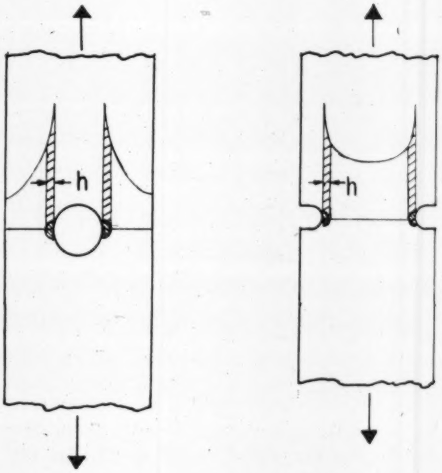

FIG. 10.—Highly Stressed Regions in Notched Plate Specimens.

Then substituting for F from Eq. 24*a* and for Δl from Eq 25, Eq 24 gives the highly stressed volume as

$$V_s \cong 0.46\sqrt{r_s b_s} t b_s \quad \text{.........(26)}$$

Similarly, in the notched specimens shown in Fig. 10,

$$V_n = 2F\Delta l \quad \text{............(24)}$$

But now

$$F \cong t2h$$

Where h (see Eq 20) is given by:

$$h = \frac{0.05}{G}$$

Substituting this expression for h gives

$$F \cong 0.1 \frac{t}{G} \quad \text{.............(27)}$$

And according to Eq 25

$$\Delta l \cong \frac{1}{\sqrt{19}} \sqrt{r_n b_n} \quad \text{........(28)}$$

then

$$V_n = 0.046 \frac{\sqrt{t r_n b_n}}{G} \quad \text{........(29)}$$

For shallow notches, G is given by Seely and Smith (6) as

$$G \cong \frac{2.5}{r_n}$$

then

$$V_n \cong 0.018\sqrt{r_n b_n}\, t r_n \quad \text{.......(30)}$$

For deep notches, G is given by Seely and Smith (6) as

$$G \cong \frac{1.9}{r_n}$$

and V_n becomes:

$$V_n \cong 0.024\sqrt{r_n b_n}\, t r_n \quad \text{.......(31)}$$

Thus, for notched plates subjected to axial load the expression for V_n is identical for both shallow and deep notches except for the numerical coefficient.

DISCUSSION

MR. BURTON HATCH.[1]—I noticed that in Fig. 7 of the paper 75 per cent of the data fell within the ±10 per cent line. Has there been any attempt to determine a distribution across those lines? Is it a normal distribution in line with the 2 σ figure mentioned, or is there any pattern at all in the data?

MR. H. T. CORTEN (*for the author*).—We did not do this determination. It is tempting to consider until you realize these data were collected over a period of years by a number of people who defined fatigue strength and endurance limit in different ways. I do not believe we could anticipate anything but a purely chance correlation of the kind you mentioned. The distribution may be normal but the source of the scatter is totally unknown and the significance would be questionable.

MR. WILLIAM N. FINDLEY.[2]—The theoretical stress concentration factor is usually defined with respect to the stress in a member having *no* stress concentration. This would correspond in Eq 7 of the paper to V_s approaching infinity, as in the pure bending of a rod of uniform cross-section. For this condition, the volume V_s in Eq 7 is indefinitely large. Therefore the ratio K_f/K_t in Eq 9 approaches zero for any size of notch—which is absurd. In view of this fact the analysis appears to break down for this special but important and readily achievable case. Thus, there would seem to be some doubt as to the validity of the basic hypothesis of a "highly stressed volume," whatever it may be. The correlation between theory and experiment reported by the author may be only a chance correlation resulting from the shape of the particular "smooth" specimen investigated.

MR. R. KUGUEL (*author*).—In answer to Mr. Findley's question regarding the application of the "highly stressed volume" approach to an infinitely large, uniformly stressed member, the author believes that it may have an academic interest, but this point of view was not considered in the paper. The "highly stressed volume" concept, as it was developed was directed toward the commonly encountered design situations.

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[2] Engineering Department, Brown University, Providence, R.I.

Moreover, the author does not think a member like that mentioned by Mr. Findley is feasible, that is, with such a shape and system of loadings to make V_s infinitely large and with no stress concentrations.

EFFECT OF OLEOPHOBIC FILMS ON FATIGUE CRACK PROPAGATION*

By W. L. Holshouser[1] and H. P. Utech[1]

Synopsis

Sharply notched rotating-beam specimens were used to evaluate the effect of oleophobic films formed by dodecyl alcohol on the rate of fatigue crack propagation in 4340 steel, 17-7 PH stainless steel, 6061-T6 aluminum alloy, and a copper-beryllium alloy (1.75 per cent Be). The number of cycles required to propagate the crack was increased by the presence of the organic compound by factors ranging from 1.4 to 5.0. This effect is attributed to the ability of compounds of this type to form films that protect the metal from oxygen and water vapor in the atmosphere.

Nearly 30 years ago Gough and Sopwith (**1**)[2] showed that the fatigue strength of metals was significantly higher in a partial vacuum than in a normal atmosphere. Later the same authors (**2**) obtained results indicating that oxygen and moisture together in the atmosphere were more damaging to copper than either component alone. Other investigators have shown comparatively little interest in the effect of the atmosphere on fatigue, and such observations as have been made were largely incidental to other investigations (**3–5**). These three references showed an improvement in fatigue properties when the specimen was protected from the atmosphere by a film of an organic liquid or grease.

Interest in these effects has been increasing in recent years, as evidenced by the work of Thompson, Wadsworth, and Louat (**6**) and of Wadsworth and Hutchings (**7**). Wadsworth and Hutchings (**7**) showed that the fatigue lives of copper and aluminum specimens were increased by factors of 20 and 5, respectively, when the air pressure was reduced from 760 to 10^{-5} mm of mercury. The fatigue life of gold, however, was not affected by reducing the air pressure.

The work of Zisman and his associates (**8**) has provided a basis for selecting organic compounds that would be expected to provide optimum protection from the atmosphere and, presumably, have a marked beneficial effect on fatigue life. They found that certain classes of polar organic compounds form tightly packed monomolecular films on metal surfaces. These films are unwetted by, or are oleophobic to, all but the lowest-boiling hydrocarbons and other nonpolar liquids. They are also highly hydrophobic.

The effect of such films on the fatigue properties of metals is being investigated at the National Bureau of Standards as part of a project sponsored by the National Aeronautics and Space Administration on the role of surface reactions in the fatigue process. Previously published results (**9**) showed that films of octyl alcohol, dodecyl alcohol, dodecylamine, and octadecylamine increased the fatigue life of smooth R. R. Moore specimens made of 4340 steel, AZ-63 magnesium alloy, and a copper-beryllium alloy (1.75 per cent Be). The films had little or no effect on similar specimens made of 6061 aluminum, 17-7 PH stainless steel, and titanium. Evidence from this and other previous investigations (5,10) indicated that in the tests of smooth R. R. Moore specimens of strong metals the crack was initiated at a late stage in the fatigue life. Thus the data presented in reference (9) were considered largely a measure of the effect of the films on crack initiation. In order to obtain similar information on crack propagation, the experiments reported below were conducted with sharply notched specimens.

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* Presented at the Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] National Bureau of Standards, Washington, D. C.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

TABLE I.—ALLOYS SELECTED FOR STUDY.

Material	Condition	Hardness
SAE 4340 steel	quenched and double tempered	32Rc
17-7 PH stainless steel	TH 1050	45 Rc
Copper-beryllium alloy (1.75 per cent Be)	solution treated and aged	36 Rc
6061 aluminum alloy	T-6	95 Brinell

MATERIALS AND TESTING PROCEDURE

The two ferrous and two non-ferrous alloys selected for study are listed in Table I. Smooth specimens of two of these materials, 4340 steel and the copper-beryllium alloy, had shown a large oleophobic film effect, while for the other two alloys the effect was small (9).

Dodecyl alcohol was selected as the film-forming material because in the previous investigation it showed a slightly greater effect on fatigue life than some of the other compounds used and more data on its effect were accumulated. It was also more convenient to use than the amines that were included in the previous work, as it is liquid at room temperature.

All of the tests were run on a rotating-beam fatigue testing machine (R. R. Moore type) operated at approximately 3000 rpm. A sketch of the specimen used is shown in Fig. 1. The 0.005-in. radius at the root of the notch was carefully ground to prevent variations in surface finish and in the amount of cold work produced in the surface.

Preliminary tests were conducted to determine the approximate number of cycles required to initiate cracks at the roots of the notches. Specimens were run for small percentages of their expected fatigue life at each stress level and then longitudinal metallographic sections were examined for cracks. The number of cycles required to produce a crack detectible at a magnification of 1000X was designated N_c, the crack propagation period, N_p, and the total fatigue life, N_f ($N_f = N_c + N_p$).

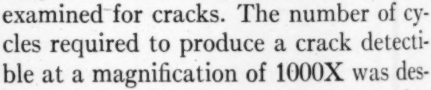

FIG. 1.—Dimensions of the Notched Fatigue Specimens.

Prior to testing, the specimens were individually cleaned by agitation in three successive containers of high-purity benzene. Except where noted otherwise in the tables, specimens to be tested in the coated condition were run for N_c cycles without coating and then, for the remainder of the fatigue life, dodecyl alcohol was dripped into the notch. Thus, the oleophobic film formed by the dodecyl alcohol was present only during the crack-propagation portion of the tests.

TABLE II.—RESULTS OF TESTS ON NOTCHED SPECIMENS TO DETERMINE THE EFFECT OF DODECYL ALCOHOL ON FATIGUE CRACK PROPAGATION.

Material	Nominal Stress Amplitude, psi	Thousands of Cycles of Stress						Ratio of N_p Values, Coated: Clean
		Clean			Coated with Dodecyl Alcohol			
		N_f[a]	N_c[a]	N_p[a]	N_f[a]	N_c[a]	N_p[a]	
4340 steel	32 500 55 000	210 33	10 2	200 31	586 63	10 2	576 61	2.9 2.0
Copper-beryllium alloy	30 000 50 000	749 27	78 3	671 24	3406 39	78 3	3328 36	5.0 1.5
6061-T6 aluminum alloy	11 500 17 000	358 81	18 4	340 77	632 120	18 4	614 116	1.8 1.5
17-7 PH stainless steel	42 500 52 500	115 32	40 12	75 20	325 39	40 12	285 27	3.8 1.4

[a] N_f = Median fatigue life (5 to 7 specimens),
N_c = Number of cycles required to produce a detectible crack, and
$N_p = N_f - N_c$ = crack-propagation portion of fatigue life.

TEST RESULTS

The portion of the total fatigue life represented by N_c for clean specimens was approximately 5 per cent for 4340 steel and 6061 aluminum, 10 per cent for the copper-beryllium alloy and 35 per cent for 17-7 PH stainless steel. These values reflect the relative resistance to crack initiation and crack propagation in air for the different materials.

The results of the tests in which dodecyl alcohol was applied only during the crack-propagation period are given in Table II. The data show that, for all four alloys, the duration of the crack-propagation period at both stress levels was increased by the presence of the oleophobic film. The magnitude of the effect is indicated by the ratios of N_p values given in the right-hand column of the table. Note also the marked effect of stress level shown for all four materials.

Some of the results on smooth specimens reported in reference (9) are given in Table III for comparison with the data on notched specimens in Table II. Although the effect of the oleophobic films on crack propagation was not completely eliminated from the data obtained on

TABLE III.—EFFECT OF OLEOPHOBIC FILMS ON FATIGUE LIFE OF SMOOTH SPECIMENS.

(Data from Frankel, Bennett, and Holshouser (9)).

Material	Stress Amplitude, psi	N_f Clean, 1000 cycles	Ratio of N_f Values, Coated: Clean
4340 steel	95 000 100 000 115 000	181 83 28	2.3 2.3 1.4
Copper-beryllium alloy	45 000 50 000 60 000	258 89 21	4.6 2.8 1.0
6061-T6 aluminum alloy	30 000 37 000	326 49	1.1 1.3
17-7 PH stainless steel	100 000 115 000	114 52	1.5 1.3

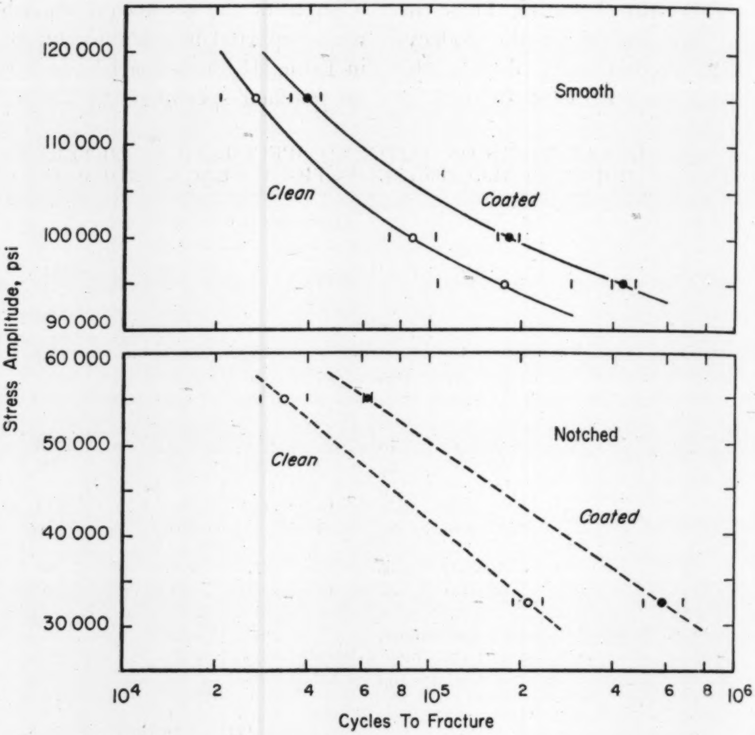

FIG. 2.—*S-N* Curves for Smooth and Notched Specimens of 4340 Steel.

Standard deviations ($\pm 1\sigma$) are also shown. "Coated" indicates that the test sections of the specimens were covered with an organic liquid that forms oleophobic films on metals.

TABLE IV.—RESULTS OF SUPPLEMENTARY FATIGUE TESTS WITH NOTCHED SPECIMENS.

(Five to 7 specimens for clean condition. Number of specimens shown in parentheses for other conditions)

Material	Nominal Stress Amplitude, psi	Median Life, thousands of cycles, for the Following Conditions of Specimens[a]		
		Clean	A	B
4340 steel	32 500 55 000	210 33	569 (2) 63 (2)	518 (2) ...
Copper-beryllium alloy	30 000	749	1776 (1)	...
6061-T6 aluminum alloy	11 500 17 000	358 81	473 (3) 104 (2)	... ...
17-7 PH stainless steel	42 500 52 500	115 32	292 (2) 54 (1)	... ...

[a] A, Coated with dodecyl alcohol prior to test. None applied during the test.

B, Coated with dodecyl alcohol prior to test. Water dripped into the notch during the entire test.

smooth specimens, this comparison does give an indication of the relative magnitude of the oleophobic film effect on crack initiation and propagation.

The data for 4340 steel are presented graphically in Fig. 2. A comparison of the vertical distance between the two curves in each set shows that the relative increase in fatigue strength due to the presence of the oleophobic film was greater for notched specimens. An appreciable difference in the dispersion of the data obtained on clean and coated specimens was noted in the previous investigation (9), the coated specimens showing considerably less scatter than the clean ones, as illustrated by the ranges of ±1 standard deviation plotted in Fig. 2. In this respect the data for notched specimens were not consistent, and no conclusion as to the effect of the coating on dispersion of the data could be drawn for any of the materials.

A few supplementary fatigue tests with notched specimens were run under the conditions noted in Table IV. In general, specimens coated with dodecyl alcohol and subsequently tested in air (condition A, Table IV) showed a somewhat smaller effect of the oleophobic film than the specimens that had the dodecyl alcohol dripped into the notch throughout the crack-propagation stage of the tests. Two 4340 steel specimens coated before testing and exposed to water dripped into the notch during the tests (Condition B, Table IV) had fatigue lives almost as long as similarly coated specimens tested in air but not exposed to water.

DISCUSSION

Oleophobic films apparently affect the fatigue process by limiting the access of molecules of oxygen or water to the metal surface. This reduces the rate of detrimental surface reactions that normally occur when specimens are stressed in air.

The difference between the effects on crack initiation and propagation may be associated with the nature of the oxide films on the metals. Detrimental surface reactions in air prior to crack initiation would be minimized by the relatively impermeable and adherent oxide film on stainless steel and aluminum. This would account for the small effect of the dodecyl alcohol on smooth specimens of 17-7 PH stainless steel and 6061-T6 aluminum alloy and the larger effect on 4340 steel and copper-beryllium, which have more permeable and less adherent films. During the crack-propagation period the protective nature of the oxide film may be relatively unimportant, as each increment of crack extension provides an area of clean metal surface which can react with any oxygen present.

While these considerations provide a possible explanation of the effects of oleophobic films on crack initiation and propagation, the mechanism of the effect of surface reactions on fatigue is still not clear. It has been suggested that, in the absence of surface reactions, the crack increment that is formed during the tensile portion of the cycle may partially weld together during the compressive half-cycle. The formation of an oxide film would inhibit this welding and thus increase the rate of crack propagation. This hypothesis does not explain the effect on crack initiation. However, the suggestion made by Thompson, Wadsworth, and Louat (6) that oxygen penetrating into slip bands can accelerate crack formation may have merit in relation to both crack propagation and initiation. This conjecture is in accord with the observations of several investigators that metal in the process of yielding under stress is more reactive than it is after the stress is removed. A clear picture of the nature of the effect of the atmosphere on fatigue will require additional experimental background.

CONCLUSIONS

The results of rotating-beam fatigue tests on sharply notched specimens of 4340 steel, 17-7 PH stainless steel, a copper-beryllium alloy, and 6061–T6 aluminum alloy appear to justify the following conclusions:

1. The rates of fatigue crack propagation in all the materials investigated were reduced by coating the specimens with dodecyl alcohol. This compound is representative of a group of polar organic compounds that form oleophobic films on metals.

2. Two materials, 6061-T6 aluminum alloy and 17-7 PH stainless steel, showed a greater oleophobic film effect with notched specimens than they had during a previous investigation with smooth specimens.

3. Notched specimens coated with dodecyl alcohol and subsequently tested in air generally showed a somewhat smaller effect of the oleophobic films than specimens that had the dodecyl alcohol dripped into the notch continually during the crack propagation stage of the tests. Exposure to water during testing did not greatly reduce the oleophobic film effect on coated specimens of 4340 steel.

Acknowledgment:

The authors wish to express their appreciation to Mr. John A. Bennett for his advice and guidance during this investigation.

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DISCUSSION

Mr. Henry E. Frankel.[1]—In Table II of the paper the values for N_c^a, the number of cycles required to produce a detectable crack, are exactly the same for the clean specimens as for the coated specimens, whereas in an earlier paper, the authors indicated that the number of cycles to produce a crack is greater with coated than with uncoated specimens.

Mr. W. L. Holshouser (*author*).—Yes, that is true because these tests were run differently from those in the first paper. The notched specimens that were run in the coated condition were not coated until a detectable crack was initiated, so that N_c^a is the same for both the clean and coated conditions. That is, we put the specimens into the machine in the clean condition and ran them long enough to produce a crack, and then started dripping dodecyl alcohol into the notch.

Mr. R. D. Behr.[2]—My first question concerns the theory that the increment of a fatigue crack which forms during the tensile portion of a cycle may partially weld together during the compression portion of the cycle in the absence of surface reactions and the authors' suggestion that oleophobic films may protect the metal from surface reactions. Would not the coatings per se prevent welding by entering the fatigue crack?

My understanding is that during the fatigue tests dodecyl alcohol was caused to drip on the specimens. Did the authors attempt to determine whether removal of heat by the alcohol contributed to the improved fatigue life of these specimens?

Mr. Holshouser.—In regard to the theory about welding, the coatings might prevent welding by preventing contact between the exposed metal surfaces. However we are not sure whether the dodecyl alcohol actually gets down into the crack during the early stages of crack formation. It may bridge over the crack and thus prevent air from getting into the crack. In this case, the dodecyl alcohol probably would not prevent welding.

In regard to the cooling effect of the dodecyl alcohol, in Table IV of the paper are shown the results of some tests run by coating the specimens before the tests were started and in which no dodecyl alcohol was dripped on the specimens during the test. The latter specimens had just about the same fatigue life as those that had dodecyl alcohol dripped on them. Therefore we feel that the dissipation of heat by the alcohol was not a factor in these experiments.

Mr. Frank Yurkoski.[3]—Realizing that these specimens were tested with smooth surfaces in one case and with controlled notches in the other, what would Mr. Holshouser's opinion be as to the effect of these coatings on weldments or other joint configurations? Is it possible that the notch effect due to a weld joint, either mechanical or metallurgical, would be so great that the film would not have any influence on it?

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[2] Research and Development Engineer, Metallurgical Laboratory, Dow Metal Products Co., Midland, Mich.

[3] Metallurgist, Research and Development Division, ACF Industries, Berwick, Pa.

MR. HOLSHOUSER.—This work on crack propagation showed that the oleophobic coating would affect the life of specimens that already had a crack in them. The specimens were cracked before the coating was dripped on. This indicates that the coatings are effective in the presence of notches and they probably would affect the fatigue life of weldments.

MR. M. R. ACHTER.[4]—It is well known that under certain conditions the life of a creep specimen is shorter in a reactive environment than in a neutral one. These creep results could not be explained by the welding hypothesis which has been advanced for fatigue. Easier crack propagation in a reactive environment has often been ascribed to weakening of the interatomic bond by reaction with a gas. This mechanism would explain both the creep and fatigue results.

It would be interesting to continue these studies using tensile fatigue instead of alternate tension and compression to eliminate the possibility of crack rewelding.

MR. HOLSHOUSER.—Yes, we do intend to do some more work on this and there are a number of experiments that might prove interesting. I appreciate your suggestions. I do not want to defend the rewelding theory but the possibility that rewelding does occur when the effect of the atmosphere is not present is frequently mentioned in discussions of the mechanism of fatigue.

MR. M. SEMCHYSHEN.[5]—It would be interesting to define precisely whether the effectiveness of oleophobic films is primarily associated with propagation or with initiation of cracks; also, whether oleophobic films would be effective in changing the transition temperature, for example, under conditions of rapid strain rates such as encountered in impact testing. As a matter of fact, perhaps the transition temperature approach might help to define the mechanism by which oleophobic films retard cracking.

MR. HOLSHOUSER.—That may be a possibility, although I believe that the tests used to determine transition temperatures are not as sensitive as fatigue tests to the condition of the surface of the specimen.

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[5] Director, Metallurgical Research, Climax Molybdenum Co., Detroit, Mich.

FATIGUE PROPERTIES OF UNCOATED AND COATED UNALLOYED MOLYBDENUM AT 1800 F, ROOM TEMPERATURE, AND -40 F*

By A. A. Mittenbergs,[1] G. D. Haley,[2] and D. N. Williams[3]

Synopsis

Fatigue behavior of unalloyed commercially pure arc-cast molybdenum was investigated in tension-tension loading on unprotected and protected, unnotched and notched sheet specimens at 1800 F, room temperature, and -40 F. Three oxidation-resistant coatings were evaluated under fatigue loading. Static tension tests were also conducted on specimens having the four surface conditions (one unprotected and three protected) at the three temperatures.

For unprotected specimens, the unnotched fatigue strength (maximum stress) at 10^7 cycles was 28,000 psi at 1800 F, 90,000 psi at room temperature, and 127,000 psi at -40 F. The notch sensitivity was low at 1800 F, but rather high at room temperature and at -40 F. All coatings lowered the fatigue strength of molybdenum somewhat at all three temperatures. The loss of fatigue strength was higher in the notched specimens.

The coatings investigated did not prove to be completely reliable for high-temperature applications under continuous fatigue loading. For limited-life high-temperature fatigue loading, however, utilization of coated molybdenum appeared to be possible with proper precautions.

In the current development of suitable materials for high-temperature service, molybdenum has been considered for some applications. Molybdenum and its alloys have been studied and developed vigorously for the past several years. Because of the poor resistance of molybdenum to oxidizing atmospheres at high temperatures, utilization of molybdenum in high-temperature applications must include, in most cases, provisions for surface protection. Therefore considerable efforts have been expended in developing suitable surface protectives, such as oxidation-resistant coatings.

Over the past several years, a certain amount of information has been developed on molybdenum and on oxidation-resistant coatings. For example, the engineering data book "Molybdenum Metal" (**1**)[4] lists over 300 references on molybdenum, its applications, properties, protection from oxidation, and other related subjects. However, fatigue information on molybdenum and its alloys is rather scarce and scattered(1–4). More information is necessary, particularly at elevated temperatures, if molybdenum is to be used as a structural material at high temperatures.

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[4] The boldface numbers in parentheses refer to the list of references appended to this paper.

For many possible applications, the feasibility of using molybdenum for high-temperature service may depend on the quality and reliability of the coatings and on their effects on the properties of molybdenum and its alloys, yet there have been no known data on fatigue behavior of coated molybdenum. This investigation was initiated to provide information on fatigue behavior of coated molybdenum as well as additional fatigue data on molybdenum.

PURPOSE AND SCOPE OF INVESTIGATION

The purpose of this investigation was to explore the fatigue behavior of molybdenum under the following variables: (1) temperature (high-, room-, and low-), (2) surface condition (uncoated and coated), and (3) stress concentration (unnotched and notched specimens).

In this program, commercial rather than special-quality materials (such as laboratory-made) were used to explore how the products of present technology may behave in service. Unalloyed molybdenum was selected to provide base-line information, since it has the lowest high-temperature strength and the lowest recrystallization temperature among commercial molybdenum-base materials. Arc-cast molybdenum was selected because it can be produced in large cross-sections and thus has potentiality for structural application.

The selection of test temperatures for the high- and low-temperature fatigue investigations was based on data obtained in studies of recrystallization and the ductile-brittle transition-temperature range, respectively, of the material received. At 1800 F, recrystallization was just starting in long-time static exposures. The test temperature of −40 F was below the transition range for the test conditions used.

In the selection of coatings, only such coatings were considered that required treatment temperatures low enough to permit their application with minimum recrystallization of the molybdenum. In addition to the unprotected molybdenum, molybdenum specimens protected by the following coatings[5] were tested:

1. Diffusion-type coating known as W-2 (a modified silicide coating).
2. Spray-type Al-Cr-Si coating. The flame spray feed contained a mixture of 20 per cent aluminum and 80 per cent chromium-silicon (40 per cent silicon) powder.
3. Nichrome-V-clad molybdenum with a chromium diffusion barrier and with specimen edges protected by Nichrome-V (80 Ni, 20 Cr) weld beads.

At 1800 F, the unprotected specimens were tested in a hydrogen atmosphere. The protected specimens were tested in air. Such a procedure permitted, at this temperature, an evaluation of both the effects of the coatings on the fatigue strength of the molybdenum and the oxidation-resistance and effectiveness of the coatings. At room temperature and at −40 F all testing was conducted in air. Fatigue tests on unnotched and on notched sheet specimens were conducted in tension-tension loading with a ratio of alternating load to mean load of $A = 0.95$. Static tension tests were also conducted at the same three temperatures on unnotched specimens having the four surface conditions (one unprotected and three protected).

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[5] The coatings tested may not have been developed or intended by the manufacturers for the conditions to which they have been subjected. Some of the coatings were not applied with the manufacturer's recommended procedures. Any failure or poor performance of a coating is, therefore, not indicative of the utility of the coating under less stringent conditions or for other applications.

MATERIAL AND PREPARATION OF TEST SPECIMENS

The material used in this program consisted of bare molybdenum and of Nichrome-V-clad molybdenum sheets. The bare sheets were hot-cold worked 75 per cent at an initial furnace temperature of 2200 F, including close to 50 per cent cross-rolling. Thereafter, these sheets were hot-roller leveled, stress relieved at 1650 for 1 hr, cleaned, and descaled. Three 0.11 by 20 by 70 in. bare sheets were used.

The clad material was produced by the roll-cladding process. Hot-rolled, recrystallized, descaled, and pickled molybdenum plate 0.40 by 10 by 35 in. was first electroplated by a commercial supplier with 3 mils of chromium per side to provide a diffusion barrier between the cladding and the base metal. Roll-cladding was done by another supplier. The plate was placed in a stainless steel frame for vacuum encapsulation. Nichrome-V sheets of 0.040-in. original thickness were placed on each side of the plate and welded to the frame under an internal, positive argon pressure. After welding, the pack was warm-evacuated, then it was rolled from a furnace temperature of 2250 F. The thickness of the pack was decreased to about one half by cross-rolling, and then the pack was direct-rolled to the final thickness of about 0.11 in. After rolling, the clad sheet was stress relieved at 1650 F for 1 hr and flattened by pressure at the end of this process. Measurements obtained on specimens of clad material indicated the following thicknesses (the over-all sheet variations may have exceeded these figures):

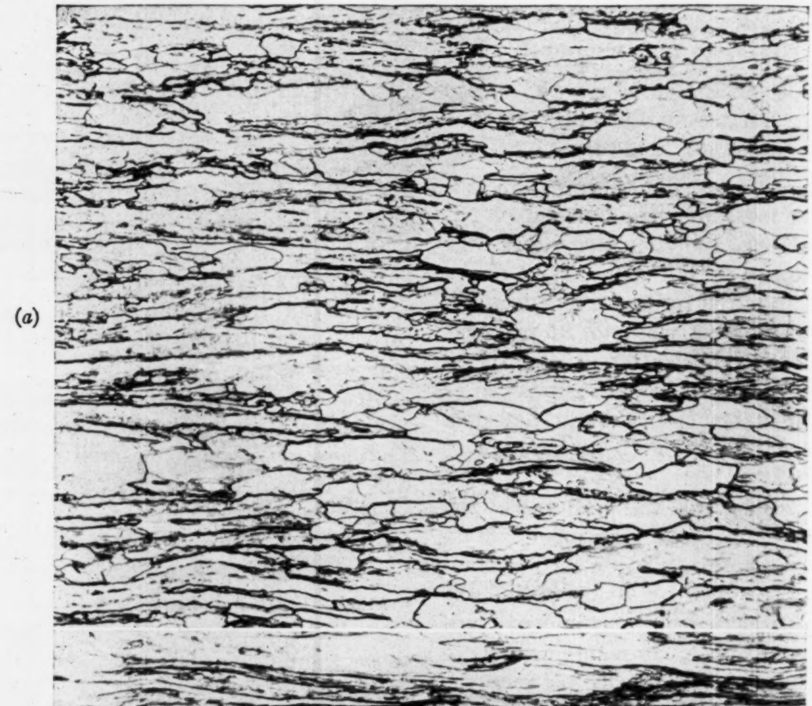
Molybdenum core..........	83.1 to 86.5 mils
Chromium barrier (each side)	0.7 to 1.0 mil
Cladding (each side)........	7.2 to 9.8 mils
Total thickness............	102 to 109 mils

The molybdenum sheet material used in this program was from two heats. As can be seen from Fig. 1(*a*), heat A exhibited slight recrystallization (about 4 per cent) in the as-received condition, whereas no recrystallization was apparent in heat B (Fig. 1(*b*)). The diamond pyramid hardness (DPH) of the as-received material was 258 (10-kg load) for heat A and 270 for heat B. The chemical analysis for both heats was 260 ppm carbon, about 10 ppm oxygen, and <0.25 ppm hydrogen.

Most of the unprotected specimens were fabricated from heat B material. The core of the clad material (Fig. 2) was also from heat B, and the coated specimens were fabricated predominantly from heat A. Since the results obtained in tension and in fatigue testing on specimens made from each heat (a number of cross-check specimens were used) did not show significant differences, the data presented in this paper do not indicate from which heat the individual specimens were made.

Fatigue specimens were machined to the dimensions shown in Fig. 3. The theoretical stress concentration factor for the notched specimens was $K_t =$ 2.0 (5). Tension specimens had a nominal test section of 0.50 by 0.09 in. and a gage length of 2 in., except for the clad specimens having the original clad-material thickness and the unprotected specimens tested at 1800 F, which had a 0.38 by 0.09 in. test section and a 2.5-in. gage length.

All specimens were cut, machined, and polished in the longitudinal direction of the sheets. To assure uniform thickness, all specimen coupons (except clad material) were surface ground using a vacuum chuck. Because of "oil can" in the received sheets and probable stress relieving caused by removal of metal, the fabricated specimens were out-of-flat, and so were the coupons of clad material.



(a) From heat A.
(b) From heat B.
FIG. 1.—Longitudinal Sections Through Molybdenum Sheets. Murakami's etch (×250).

Since out-of-flatness could influence the fatigue test results, all specimens were creep flattened at 1700 F for 4 hr in hydrogen. Experiments conducted on specimens of the material showed that the creep flattening process did not cause any recrystallization, but only a decrease in hardness by an average of 10 DPH units for the bare specimens. The core of the clad material remained completely unrecrystallized, and its diamond pyramid hardness of about 253 (10-kg load) did not change. After creep flattening, the bare specimens were essentially flat, but the clad specimens still had some out-of-flatness.

Subsequent to creep flattening, all specimens were cleaned and the fatigue specimens were polished. Since coating processes generally require that there should be no sharp edges in the coated sections, all edges within the test sections were rounded with about 0.01 in. radius. Surface roughness was generally 4 to 6 rms in the longitudinal direction and 7 to 10 rms in the transverse direction for

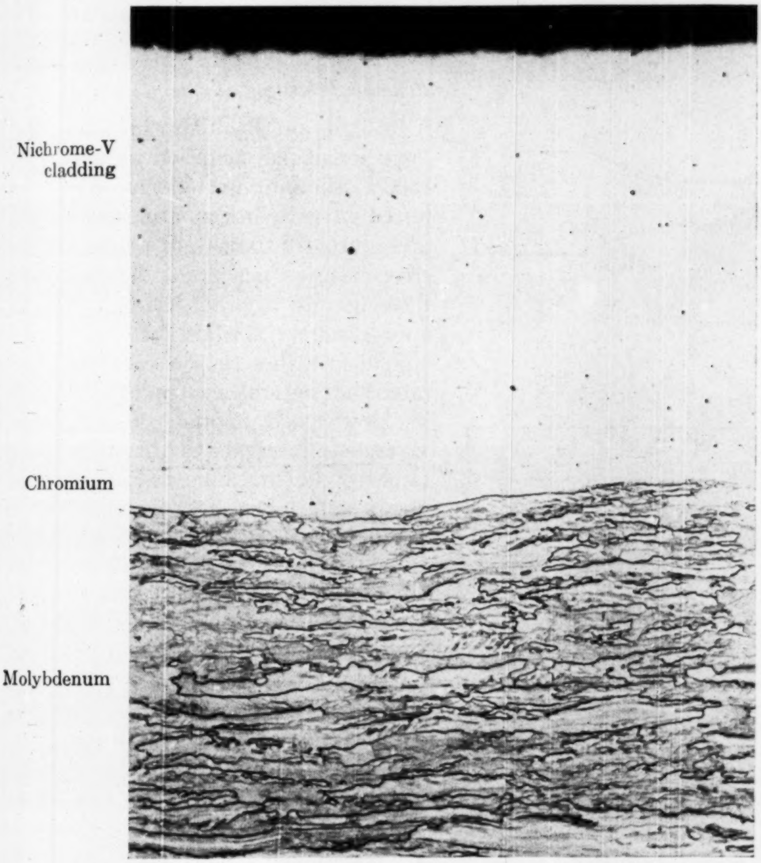


FIG. 2.—Longitudinal Section Through Clad Material as Received. Murakami's etch (×250). Note good bonding between molybdenum and chromium and between chromium and Nichrome-V.

both the surfaces and the edges. The surfaces of clad specimens were left as received (the edges were polished).

The W-2 and Al-Cr-Si coatings were applied on machined and polished test specimens by commercial suppliers. The edges of the clad specimens were protected with Nichrome-V weld beads. Representative microstructures of the two coatings on molybdenum and a Nichrome-V weld on a specimen edge are shown in Figs. 4 to 6 (see Fig. 2 for microstructures of clad material). The thickness of the W-2 coating varied between 1.2 and 2 mils and that of the Al-Cr-Si coating between 4 and 25 mils. Both coating processes caused some recrystallization, so that the base metal was recrystallized 5 to 10 per cent (with a hardness drop to 242 DPH) in the W-2-coated specimens and about 5 per cent (with a hardness drop to 234 DPH) in the Al-Cr-Si-coated specimens. Also, a number of specimens had become out of flat during coating. The final out-of-flatness of protected specimens varied between 0 and 35 mils in an 8-in. length. No correlation, however, was found between this out-of-flatness and the fatigue test results.

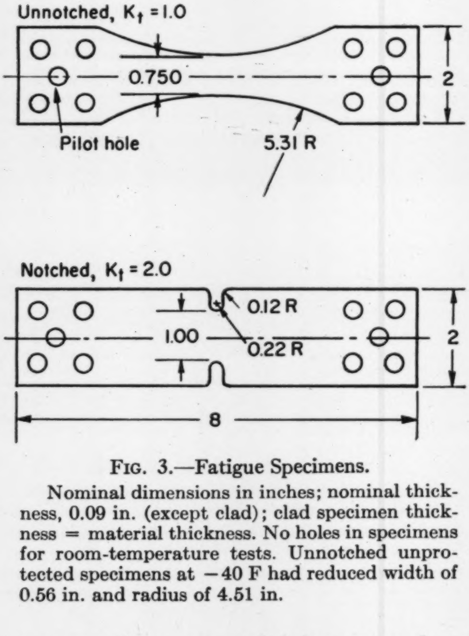


FIG. 3.—Fatigue Specimens.

Nominal dimensions in inches; nominal thickness, 0.09 in. (except clad); clad specimen thickness = material thickness. No holes in specimens for room-temperature tests. Unnotched unprotected specimens at −40 F had reduced width of 0.56 in. and radius of 4.51 in.

TEST PROCEDURES AND EQUIPMENT

Tension Testing:

In high- and low-temperature testing, 4 specimens per condition were used. At 1800 F, the unprotected specimens were tested in a hydrogen atmosphere. The procedures for these tests, performed in a creep-testing machine, differed from those used in all other testing in that an approximately constant loading rate was employed rather than a constant strain rate. Loading was accomplished by adding preweighed amounts of steel pellets at regular intervals to the weight container of the machine. Strain measurements were taken after each addition of weights from a platinum-strip extensometer attached to the specimens. The average strain rate was 0.0006 to 0.0011 in. per in. per min until the 0.2 per cent offset yield strength, and it increased progressively thereafter so that the overall strain rate to rupture was between 0.007 and 0.014 in. per in. per min. The temperature was checked with three thermocouples attached to the center and to both ends of the test section.

The protected specimens were tested at 1800 F in air in an electric-resistance furnace. Temperature was checked with three thermocouples as above. For two specimens of each condition, a constant crosshead speed was used that corresponded to about 0.003 in. per in. per min strain rate at the test section at the beginning of the tests. On the other two specimens, the crosshead speed was increased to give a strain rate of about 0.06 in. per in. per min (if related to the initial period of test) after the 0.2 per cent offset yield strength was reached. The latter specimens gave about 10 per cent (average) higher ultimate tensile strength.

Room-temperature tension testing was conducted at a constant head speed that corresponded to a strain rate of 0.003 in. per in. per min in the test section of the specimens at the beginning of the tests. Three to six specimens were used per condition.

Tension testing at −40 F was performed with the specimens placed in an alcohol and dry ice bath. The temperature was checked with a thermometer. A strain rate of 0.003 in. per in. per min was used throughout these tests.

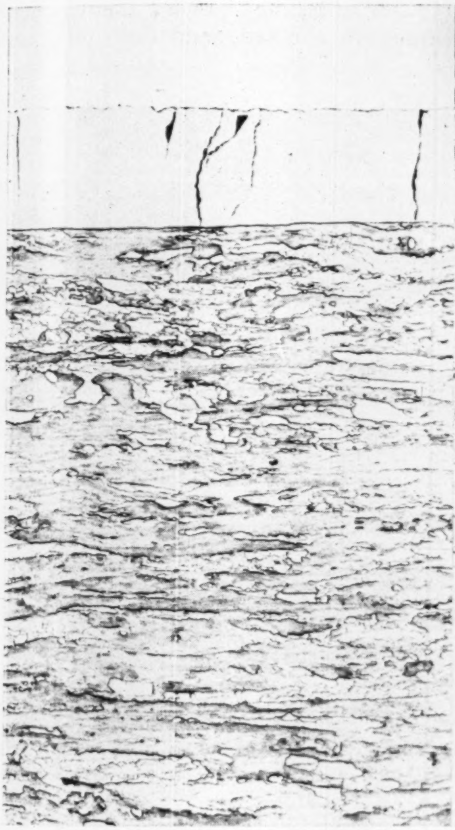


FIG. 4.—Longitudinal Section Through W-2-Coated Molybdenum as Received. Murakami's etch (×250).

Note cracks through the coating.

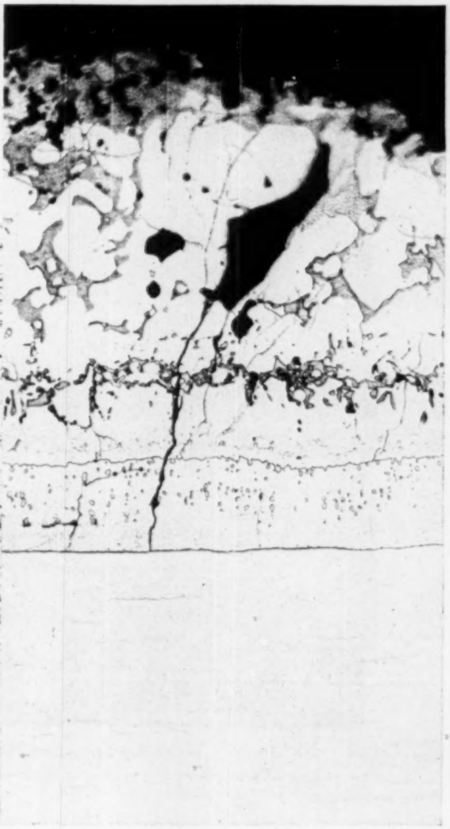


FIG. 5.—Longitudinal Section Through Al-Cr-Si-Coated Molybdenum as Received. Murakami's etch (×250).

Note multiphase nature of the coating, the distinct layer structure, and the cracks through the coating.

Fatigue Testing:

Testing at 1800 F was performed in an Ivy axial-loading fatigue machine that operates at a constant speed of 3600 cpm and is basically a constant-force machine. A Lepel 5-kw output, 250- to

600-kc induction-heating unit provided the 1800-F specimen test temperature. Although this temperature could have been attained with resistance furnaces, induction heating was chosen in order to develop techniques and procedures for future fatigue studies at still higher temperatures that could not be attained with resistance furnaces.

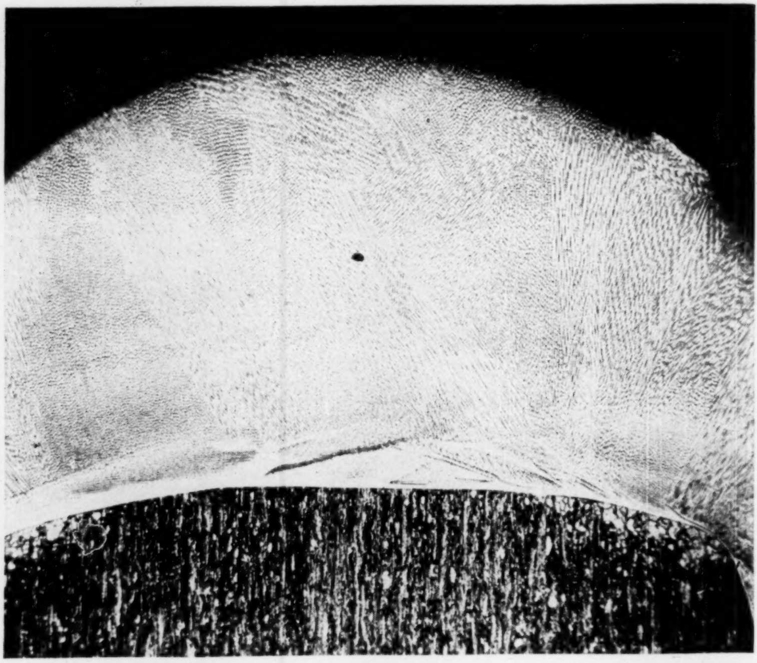


FIG. 6.—Nichrome-V Weld Bead on Clad Molybdenum Specimen. Murakami's etch (×50).

Note rounding of molybdenum edges, indicating solution of molybdenum, and recrystallization at these locations.

The induction coils were made of copper tubing and had four to six windings. They had an elongated shape, the center portions of the longer sides being bent slightly toward the center. Such a coil shape provided rather uniform temperature distribution across the specimens within the test sections. In the longitudinal direction of the specimens, the uniformly heated length in which the test temperature differential did not exceed about 10 F was between 0.75 and 1.00 in. Outside this length a temperature gradient existed. The high thermal conductivity of molybdenum was apparently contributing to the relatively uniform temperature distribution within the test section.

During testing at 1800 F the temperature was checked periodically with an optical pyrometer. The required reading (apparent temperature) was determined for each surface condition through temperature-calibration experiments employing thermocouples. For the unprotected specimens the apparent temperature was also determined from the emissivity value for molybdenum surfaces. Good agreement was obtained between the two methods. During fatigue tests the temperature at the specimen test sections was monitored manually by adjusting the power control of the induction heating unit. Such a method required close attention at the beginning of each test until an equilibrium condition was reached. It was estimated that the temperature within the test sections of the specimens was maintained within 1800 ± 25 F limits.

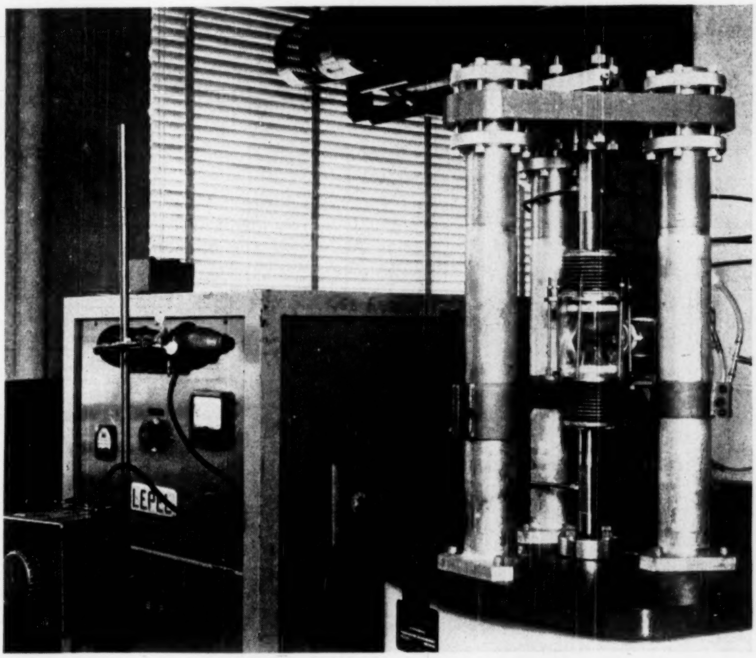


FIG. 7.—Upper Portion of Ivy Machine with a Mounted Specimen Within the Hydrogen Envelope, Induction Heating Unit, and Optical Pyrometer.

The unprotected molybdenum specimens were tested within a hydrogen-atmosphere envelope as shown in Fig. 7. This envelope consisted of a Vycor glass shell held between two units of brass bellows and flanges mounted around grips. Silicon-rubber seals were used at both ends of the shell and O-rings served as seals at the grips. All flanges were water-cooled. Gas passages were provided through the grips. Prior to and after the feeding of hydrogen, the system was purged with argon. Both gases were fed with a slight pressure and escaped through a liquid (mineral oil) trap against an electric heating coil that caused the hydrogen to burn. The same pair of grips and the same general arrangement were used for testing coated specimens in air, but, of course, without the envelope.

The room temperature fatigue tests were conducted on a 6-ton axial-loading Schenck fatigue machine. The frequency of the cyclic load varied between 2000 and 2300 cpm, depending on the load level.

Testing at -40 F was performed on another Ivy machine with a cooling unit placed on it. The scheme of the cooling unit, shown in Fig. 8, is self-explanatory. A thermocouple in the air stream in front of the specimens monitored the system by switching the liquid pump on and off. The temperature-distribution pattern in the test specimens and the air temperature required to provide the −40-F testing temperature at the center of the specimens were determined experimentally by means of specimens instrumented with thermocouples. The temperature fluctuations due to cycling of the cooling unit were within ±3 F. The air temperature settings were between −44 F and −46 F (depending on load level), and the temperature at the test sections of the specimens was maintained within estimated −40 ±5 F limits.

DISCUSSION OF RESULTS

The fatigue test results are plotted in Figs. 9(*a*) to (*d*). The maximum stresses at endurance limit (at 10^7 cycles for 1800 F) are listed in Table I together with the average tension data obtained. It should be emphasized that in tension testing molybdenum was found to be strain-rate sensitive at all three temperatures, and, therefore, these data should be interpreted and used with caution. Because of the exploratory nature and the limited scope of this program, the strain-rate effects were not investigated in detail nor determined quantitatively.

As can be seen from the table, the coatings generally lowered the ultimate tensile and yield strengths of the molybdenum specimens. Clad specimens tested at room temperature, with the stresses related to the molybdenum core only, were exceptions. The high ultimate tensile and yield strengths of clad molybdenum at room temperature might be attributable to the effect of the different rolling schedule for clad material and to the strengthening effect of the cladding. The ductility of the clad edge-welded specimens at room temperature was quite low, and these specimens failed in a brittle manner, apparently because of the notch effects resulting from the presence of the relatively brittle edge welds. Clad material with no edge welds exhibited considerable ductility and higher ultimate tensile and yield strengths (see Table I) than edge-welded specimens.

The fatigue fractures at 1800 F were generally of mixed nature (see Fig. 10), with a certain amount of brittle fracture (caused by crack initiation and propaga-

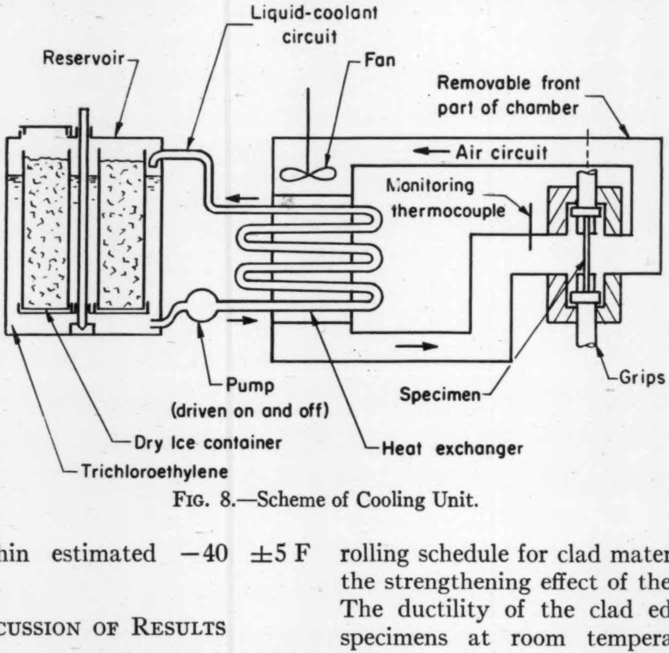


FIG. 8.—Scheme of Cooling Unit.

tion). The remaining cross-section failed in a ductile manner. The amount of brittle fracture generally increased with decreasing stress level (increasing life-time), being considerably higher in the notched specimens. The protected specimens exhibited less ductility than the unprotected. The amount of brittle fracture varied between almost zero (unnotched unprotected specimens at high stresses) and 100 per cent (notched W-2-coated specimens at low stresses).

During high-temperature testing, the W-2 coating appeared to be uniformly protective. No premature failures caused by coating failure and oxidation were experienced. On one unnotched long-life specimen a deep pit was formed by oxidation at one edge near the test section shortly before failure. This specimen failed, however, through the unoxidized test section. Of the Al-Cr-Si-coated specimens, one unnotched and four notched specimens failed prematurely because of coating failures and oxidation. Figure 11 shows one of these notched specimens. Of the clad specimens, one unnotched (shown in Fig. 12) and one

TABLE I.—TENSION[a] AND FATIGUE[b] TEST RESULTS.

Temperature, deg Fahr	Ultimate Tensile Strength, psi	Yield Strength, 0.2 per cent Offset, psi	Elongation, per cent	Fatigue Strength,[c] psi	
				Unnotched	Notched
Unprotected Specimens					
1800	52 000	44 000	9	28 000	26 000
Room temperature	105 000	99 000	26	90 000	55 000
−40	121 000	[d]	0.06[e]	127 000	78 000
W-2-Coated Specimens[f]					
1800	46 000	42 000	12	26 000	15 000
Room temperature	94 000	88 000	31	68 000	40 000
−40	85 000	[d]	0[e]	80 000	40 000
Al-Cr-Si-Coated Specimens[f]					
1800	42 000	38 000	14	23 000	14 000
Room temperature	89 000	80 000	31	76 000	42 000
−40	119 000	[d]	0.10[e]	98 000	49 000
Clad Specimens[g]					
1800	48 000	43 000	14	26 000	18 000
Room temperature	112 000	108 000	1.6	67 000	17 000
	124 000[h]	116 000[h]	26[h]	...	...
−40	69 000	[d]	0[e]	74 000	43 000

[a] Average values of several specimens.

[b] In tension-tension loading with a ratio of alternating load to mean load of $A = 0.95$.

[c] "Fatigue strength" as given in the table is the maximum stress at 10^7 cycles for 1800 F and at endurance limit for room temperature and for −40 F.

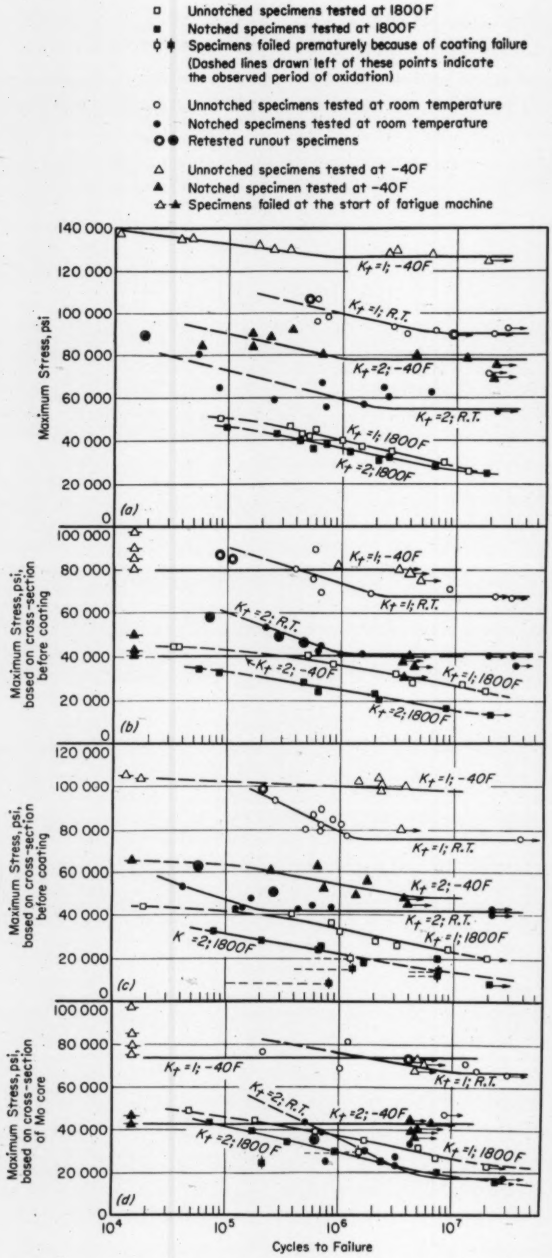
[d] Specimens failed before reaching 0.2 per cent offset yield strength.

[e] Determined from stress-strain recordings.

[f] Based on specimen cross-section before coating.

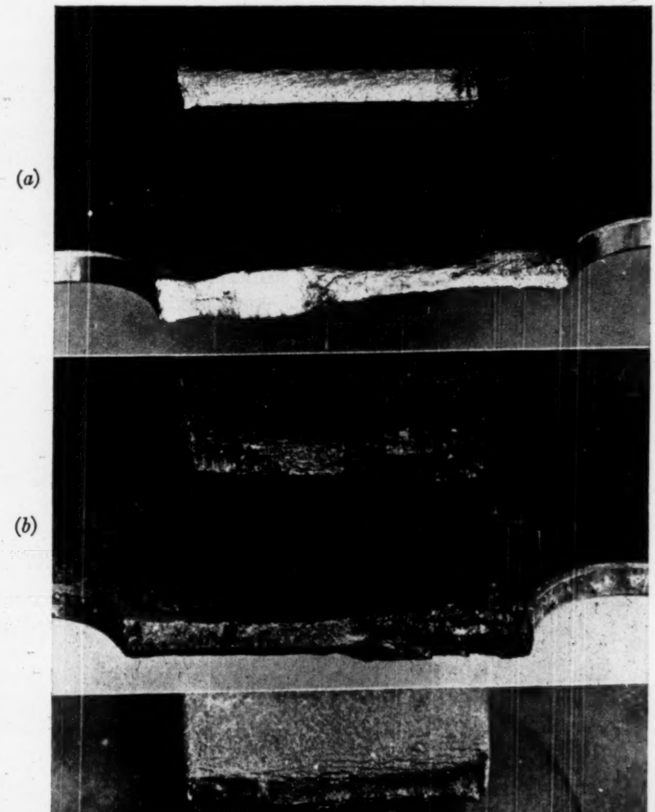
[g] Based on cross-section of the molybdenum core only.

[h] These data are for clad material with no edge welds as determined by the supplier at room temperature.



(*a*) Unprotected specimens.
(*b*) W-2-coated specimens.
(*c*) Al-Cr-Si-coated specimens.
(*d*) Clad specimens.

FIG. 9.—Fatigue Test Results.



(*a*) Unprotected (tested in hydrogen).
(*b*) W-2-coated (tested in air).
(*c*) Al-Cr-Si-coated (tested in air).
(*d*) Clad (tested in air).

FIG. 10.—Typical Fractures of Fatigue Specimens Tested at 1800 F.

notched specimen failed prematurely because of defects in the edge welds and oxidation occurring under the cladding.

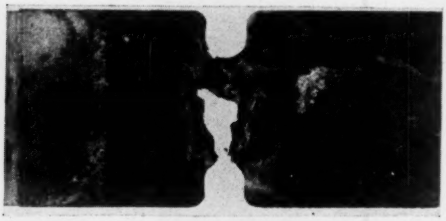


FIG. 11.—Notched Al-Cr-Si-Coated Fatigue Specimen, Tested at 1800 F in Air, That Failed Prematurely Because of Oxidation.

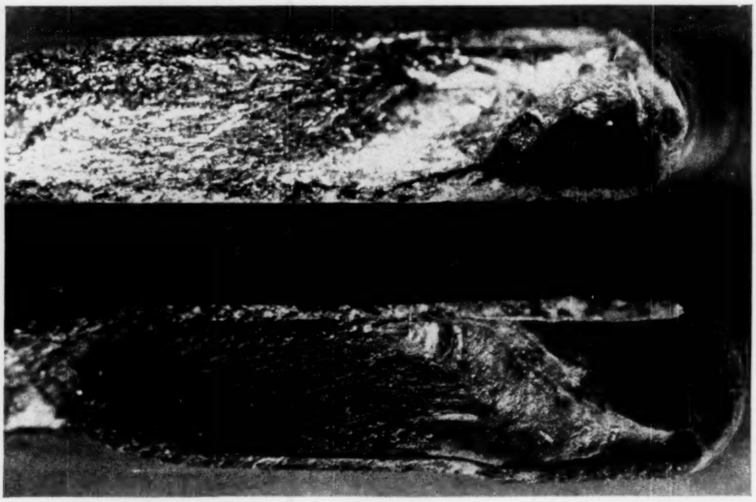


FIG. 12.—Fracture of an Unnotched Clad Specimen, Tested at 1800 F in Air, That Failed Prematurely Because of Oxidation.

At room temperature and at −40 F, the fractures of fatigue specimens were generally of a brittle nature (Fig. 13), with occasional traces of lamination. In many specimens cracks were present in the vicinity of the fractures, caused possibly by the shock at failure. A number of specimens tested at −40 F failed into more than two pieces. Most of the unnotched clad specimens tested at room temperature broke off-center, indicating probable notch effect from edge welds. The adherence of the W-2 coating appeared to be excellent at both room temperature and −40 F. The adherence of the Al-Si-Cr coating was considerably weaker. This coating came off near the fractures at failure on most specimens. The adherence of the cladding appeared to be very good.

In high-temperature fatigue test data, the scatter was not large, except for the Al-Cr-Si-coated specimens, for which the scatter apparently was increased by the large variations observed in the coating thickness. The higher scatter in room- and low-temperature test data probably is attributable to the rather brittle nature of the uncoated and coated molybdenum at these temperatures. For protected specimens, the coating variations and metallurgical notch effects from the coatings and additional effects from edge welds in clad specimens probably contributed to the scatter.

All fatigue curves obtained at 1800 F were somewhat similar in nature, having

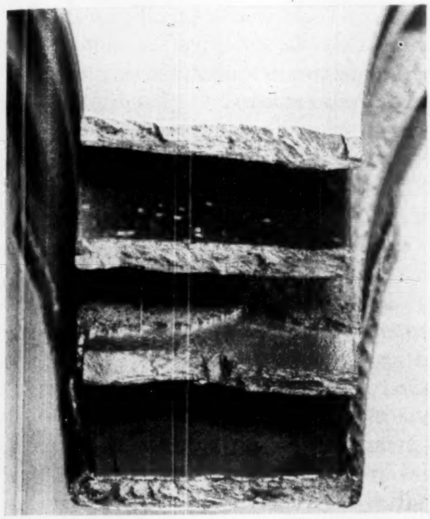


FIG. 13.—Typical Fractures of Unnotched Fatigue Specimens Tested at Room Temperature.

From top: Unprotected, W-2-coated, Al-Cr-Si-coated, and clad. (Appearance of fractures of notched fatigue specimens and those tested at −40 F is very similar to that shown in this picture.)

approximately similar slopes. Endurance limits, if such existed at this temperature, probably would have been reached at considerably longer lifetimes and lower stresses than those used in this program. Some indications were obtained from the test points that a "knee" may exist in the fatigue curves at about 2×10^7 cycles. In the short-life range, a normal trend to flatten out was indicated for the *S-N* curves. Fatigue curves obtained at room temperature and at −40 F indicated endurance limits.

The fatigue curves obtained at −40 F proved to be very flat, which is characteristic of brittle materials. The unprotected and Al-Cr-Si-coated specimens exhibited a very slight ductility at −40 F in tension testing and had flat but normal fatigue curves. On the other hand, the W-2-coated and the clad specimens exhibited completely brittle behavior in tension testing at −40 F and their fatigue strength at −40 F was independent of lifetime. The fatigue behavior of the latter two groups of specimens was peculiar in that the specimens either failed when the fatigue machine was started or ran out. One unnotched W-2-coated specimen which failed at about 940,000 cycles was an exception. Therefore, the *S-N* curves for W-2-coated and the clad specimens at −40 F could be represented by horizontal lines.

As can be seen from Table I, the maximum stress at endurance limit for the unnotched unprotected and clad specimens tested at −40 F was higher than the ultimate tensile strength at this temperature. In some other cases, at all temperatures (see *S-N* curves) the short-life fatigue strength (maximum stress) of unnotched specimens was higher than or close to the ultimate tensile strength of specimens of the same surface condition tested at the same temperatures. Such behavior apparently was caused by the effect of the much higher strain rates used in fatigue testing. When the short-life fatigue strength was lower than the ultimate tensile strength, mainly in protected specimens, the strain-rate effect was probably overshadowed by the metallurgical notch effects present in the protected specimens.

For unprotected specimens, the unnotched fatigue strength (maximum stress) was 28,000 psi at 1800 F (10^7 cycles), 90,000 psi at room temperature, and 127,000 psi at −40 F. All coatings lowered the fatigue strength of molybdenum at all three temperatures. The loss in fatigue strength of protected molybdenum varied at the various test conditions. For unnotched specimens tested at 1800 F, the decrease in fatigue strength of protected specimens was relatively small. The decrease in fatigue strength of unnotched protected specimens at room temperature and particularly at the low temperature was considerably larger.

The notch sensitivity of unprotected molybdenum tested at 1800 F was low; the value of the fatigue notch factor (ratio of unnotched fatigue strength to notched) was only 1.08 as compared with the theoretical stress concentration factor of 2.0 for the notched specimens. At the high temperature, all coatings considerably increased the notch sensitivity of molybdenum. At both room temperature and −40 F, the fatigue notch factor for unprotected molybdenum was about 1.63. For protected molybdenum, the fatigue notch factor was higher in all cases. In some of these, the increase in fatigue notch factor was slight. For W-2- and Al-Cr-Si-coated notched specimens tested at −40 F, the fatigue notch factor was the same (2.0) as the theoretical stress concentration factor. For the clad notched specimens tested at room temperature, the fatigue notch factor was close to the value of 4.0 (double the theoretical stress concentration factor). Such a result indicated that the metallurgical notch effects and particularly the effect of edge welds contributed considerably to the stress concentration at the notches.

It is interesting to note that the W-2-coated and the clad specimens, which at high temperature had less loss of fatigue strength than the Al-Cr-Si-coated specimens, had higher losses in fatigue strength at room temperature and at −40 F. Generally, the decrease in fatigue strength of protected specimens may have had several causes, with the metallurgical notch effects created by diffusion on all coatings and additional effects from edge welds on clad specimens probably being the major factors. The slight recrystallization in the W-2- and Al-Cr-Si-coated specimens that occurred during the coating processes may have been another factor. The treatments used during the coating processes may have had some additional effects. The out-of-flatness of coated specimens may have been still another factor, although no correlation could be detected between the out-of-flatness and the fatigue results. The loss of fatigue strength in notched specimens was greater than in unnotched specimens in all cases, indicating that the coatings are more detrimental in applications where geometric discontinuities exist.

From the viewpoint of coating protectiveness at high temperature, none of the coatings could be considered completely reliable. The W-2 coating seemed to be the best of the three. The oxidation protectiveness in clad material appeared to be quite good generally. However, protection of the edges seemed to be a critical item, since two clad specimens failed prematurely because of edge-weld defects.

It appears that further developments and improvements in oxidation-resistant coatings are necessary for successful and reliable utilization of coated molybdenum and molybdenum-base alloys in applications of continuous fatigue loading at high temperatures. The potential of molybdenum for high-temperature applications lies in the molybdenum-base alloys that have higher strength at elevated temperatures and have higher recrystallization temperatures. For service temperatures higher than that used in this program, the requirements for the coatings under fatigue loading would be even more severe.

CONCLUSIONS

1. Molybdenum was found to be strain-rate sensitive at all three test temperatures.

2. For unprotected molybdenum, the unnotched fatigue strength (maximum stress) at 10^7 cycles was 28,000 psi at

1800 F, 90,000 psi at room temperature, and 127,000 psi at −40 F.

3. The notch sensitivity of unprotected molybdenum was low at 1800 F, but rather high at room temperature and −40 F.

4. All coatings lowered the fatigue strength of molybdenum. However, the loss of fatigue strength in protected unnotched specimens at 1800 F was small.

5. The coatings increased the notch sensitivity in all cases; thus they were more detrimental to the fatigue strength of notched specimens than to that of unnotched specimens.

6. The coatings investigated did not prove to be completely reliable for high-temperature applications under continuous fatigue loading. For limited-life high-temperature fatigue loading, utilization of protected molybdenum appeared to be possible with proper precautions.

7. The results of this investigation indicated the potentiality of protected molybdenum for high-temperature structural applications. However, additional research and development appeared to be necessary, particularly of the coatings, to increase their reliability under fatigue loading.

Acknowledgment:

This research was supported in whole by the Air Research and Development Command, United States Air Force, under Contract No. AF 33(616)-5915, monitored by the Materials Central, Directorate of Advanced Systems Technology, Wright Air Development Division, Wright-Patterson Air Force Base, Ohio. The program is described in more detail in WADD Technical Report 60-427 entitled "Investigation of the Fatigue Properties of Molybdenum Under Various Conditions of Temperature, Coatings, and Stress Concentration," dated Oct., 1960. Among those who coöperated in this research were Messrs. W. J. Trapp and D. M. Forney, Jr., of WADD Materials Central, and Messrs. L. R. Jackson, H. J. Grover, R. I. Jaffee, A. C. Koppenhafer, A. F. Leatherman, and C. F. Speich of Battelle Memorial Inst.

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(4) R. G. Frank and W. F. Zimmerman, "Materials for Rockets and Missiles," The MacMillan Co., New York, N. Y. (1959).

(5) R. E. Peterson, "Stress Concentration Design Factors," John Wiley and Sons, Inc., New York, N. Y., 1953, p. 27.

DISCUSSION

MR. GEORGE GOHN.[1]—I wonder if, when tests are made at these high temperatures (1800 F), the stress concentration estimated by the authors really exists after the first few cycles, or if there is not stress relief because the material is stressed well above the elastic limit? If so, then we have two different types of test specimens in which no stress concentration is present. I am raising this question as a possible explanation for the close agreement between the tests on notched and unnotched specimens tested at 1800 F and the marked difference in test results at room temperature and at −40 F.

MR. A. A. MITTENBERGS (*author*).—I think this is a good point. Generally, we did find that the notch effect at the high temperature was lower than at room temperature or −40 F, and the stress relief is probably the cause. We did not investigate any of the details.

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[1] Supervisor, Mechanical Testing Laboratory, Bell Telephone Laboratories, Inc., New York, N. Y.

HIGH-TEMPERATURE FATIGUE AND BENDING STRAIN MEASUREMENTS IN CONTROLLED ENVIRONMENTS*

By G. J. Danek, Jr.,[1] H. H. Smith,[1] and M. R. Achter[1]

Synopsis

Fatigue properties in air and in vacuum of nickel, type 316 stainless steel, and Inconel X at 1500 F are compared. Sheet metal specimens were cycled in reversed bending at their resonant frequency by means of a magnetic coupling. Since at this temperature all of these materials, except Inconel X, deform plastically, an optical technique for measuring plastic bending strain at elevated temperature has been developed.

The effect of environment on fatigue properties is similar for all three materials. At high strains, fatigue lives are superior in vacuum, and with decreasing strain the vacuum and air curves converge. With nickel, tested in thinner sections, the fatigue curves cross to produce a reversal in the atmospheric effect at low strains. The results of this investigation can be explained by a mechanism previously proposed to interpret similar findings in creep, which involves the simultaneous operation of two competing processes. The effects of section size and vibration frequency are discussed in terms of the proposed mechanism.

Some implications of these results to design of high-temperature structures are presented.

The requirements in many current applications that materials operate under cyclic stresses in environments other than air has stimulated work on the effect of atmosphere on fatigue strength. Two basic methods have been used to transmit motion through the wall of an environmental chamber by the investigators in the field. Gough and Sopwith (**1**)[2] performed room-temperature axial fatigue tests in vacuum using a flexible bellows as an integral part of the chamber. Improvements over room-temperature fatigue limits in air are reported of 5 per cent, 13 per cent, and 26 per cent for mild steel, copper, and brass, respectively, when the tests are performed in vacuum pressures of 5×10^{-4} to 1×10^{-3} mm of mercury. A negligible environmental effect was found for a copper-nickel alloy and a nickel-chromium steel. The bellows seal principle was also used by Stephens and Sinclair (**2**) to test thin-wall molybdenum in vacuum under repeated axial load at 1700 F. In both investigations, since the load was applied outside the vacuum chamber allowance had to be made for the bellows seal if light loads were used. Wadsworth and Hutchings (**3**) used external electromagnets, pulsing at a rate dictated by the alternating current, to conduct room-temperature, reverse-bending fatigue tests at resonance. Surface strain was determined by calibration runs with strain

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* Presented at the Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] Metallurgy Division, U. S. Naval Research Laboratory, Washington, D. C.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

gages. They reported that the ratio of fatigue life in air to that in a vacuum of 10^{-5} mm of mercury was 1:20 for copper, 1:5 for aluminum, and 1:1 for gold. Snowden (4) also reported longer lives in a vacuum of 10^{-6} mm of mercury for reverse-bending fatigue at room temperature. The ratio of life in vacuum to that in air was 6 for aluminum and as much as 50 for lead.

In contrast to these results at room temperature, published data on the high-temperature atmosphere effect, confined to creep-rupture testing, have mostly shown higher strengths in air than in an inert gas. Shahinian and Achter (5,6), however, have demonstrated that with a sufficiently large range of variation of test conditions the effect of atmosphere may be reversed. At low temperature and high stress, the creep strength of nickel and nickel-base alloys is greater in vacuum, while at low stress and high temperatures it is greater in air. A mechanism to explain this reversal was proposed based on two competing processes. Adsorption of gases lowers surface energy and reduces the work required to propagate a crack, while oxidation can increase the hardness and strength.

To determine whether this reversal of the atmosphere effect would be observed in high-temperature fatigue, testing equipment and a technique for measuring bending strain were developed. The results of studies on nickel, type 316 stainless steel, and Inconel X are discussed in terms of the proposed mechanism.

EXPERIMENTAL PROCEDURE

Test Equipment:

Equipment previously developed for high-temperature, controlled-environment, flexural fatigue tests (7) was modified by the addition of a servomechanism to hold amplitude constant (8). An assembly consisting of a sheet metal specimen, extension rod, and permanent magnet is driven by two external electromagnets, tuned to resonance. At either extreme of its excursion, the extension rod strikes an electrical contact, causing a signal to be sent to a servomechanism which holds amplitude constant within 0.3 per cent. When a crack begins to propagate, the servomechanisms drive upscale to satisfy the increasing power requirement, close a limit switch, and end the test.

Testing Conditions:

Specimen temperature, measured by a platinum–platinum-rhodium thermocouple at the lower end of the reduced section, was maintained at 1500 ± 2 F. A conventional mechanical and diffusion pump combination was used to produce starting vacuum pressures ranging from 2×10^{-6} to 1×10^{-5} mm of mercury. Fatigue tests were conducted in air and in vacuum on 0.050 in. thick type 316 stainless steel and Inconel X at 5 cps. Nickel 0.035 in. thick was vibrated at 2 cps and 0.060 in. thick nickel at 5 cps; to investigate size and frequency effects nickel 0.118 in. thick was vibrated at 5 and 12 cps. Heavy tuning weights were used to reduce the resonant frequency of the thick nickel.

Specimen Preparation and Materials:

Details of specimen geometry have been reported previously (7). The width at the minimum section was 0.187 in. and the thickness was chosen to produce the desired test frequency. The nickel had a purity of 99.8 per cent and contained chromium, iron, magnesium, silicon, and carbon as impurities. It was supplied in the form of $\frac{1}{8}$ in. thick sheet which was cold rolled to within 0.012 in. of the thickness of the two thinner gages. The alloy specimens were machined from $\frac{1}{16}$-in. sheet. The Inconel X was heat treated for 2 hr at 2100 F, 24 hr at 1550

F, and 20 hr at 1300 F before machining. All materials were electropolished to a tolerance of ±0.0001 in. before test. To determine whether the vacuum atmosphere was affecting fatigue strength by removing gases and other volatiles, several tests were run in air on nickel and stainless steel which had been first vacuum annealed at 1500 F for 16 hr. Within experimental error, there was no effect of the prior vacuum anneal.

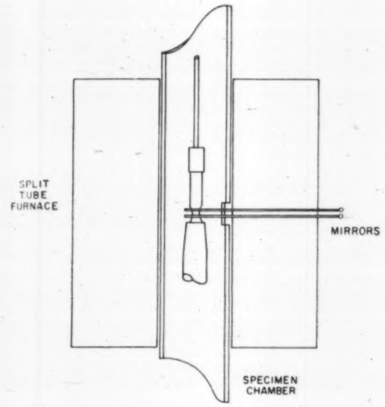


Fig. 1.—Mirrors Fixed to Specimen for Optical Measurement of Strain.

Strain Measurements:

At 1500 F, the nickel and stainless steel deform plastically, necessitating the measurement of strain instead of stress. Stress calculations by a method previously described (7) are valid only in the elastic range. An optical technique for measuring bending strains was used which is based upon the determination of the bending radius of curvature. Two mirrors are arranged symmetrically about the reduced section and parallel to the face of the specimen by means of spot-welded rods (Fig. 1). These mirrors oscillate as the specimen is cycled in reversed bending and reflect streaks of light to a screen. Streak lengths measured from photographs are used to compute curvature and surface strain.

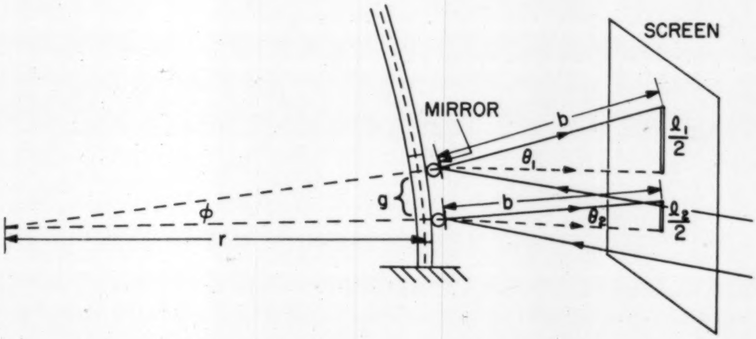
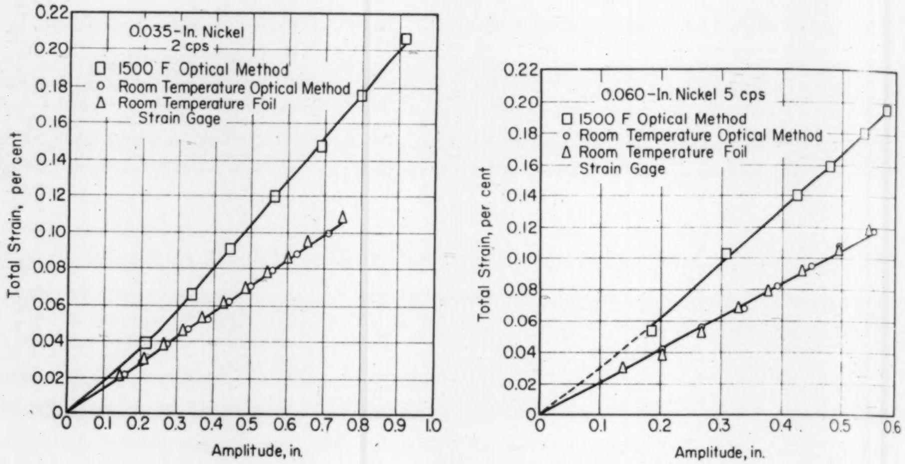


Fig. 2.—Schematic Diagram of Arrangement for Optical Measurements of Strain.

The derivation of the relationship used to compute surface strain is based on the assumption of circular bending in the gage length, in this case 0.250 in. Figure 2 depicts schematically the streaks of light traced on a screen after bending through one-half amplitude. The uncorrected strain, ϵ_0, is given by

$$\epsilon_0 = \frac{d}{r} \quad \text{(1)}$$

where d is the half thickness and r is the radius of curvature. This relationship is valid if the strain gradient is linear. Since, at the amplitudes used, there is no permanent change of shape of the reduced



(*a*) 0.035 in. thick nickel cycled at 2 cps. (*b*) 0.060 in. thick nickel cycled at 5 cps.

FIG. 3.—Strain Measurement Calibration Curves for Nickel.

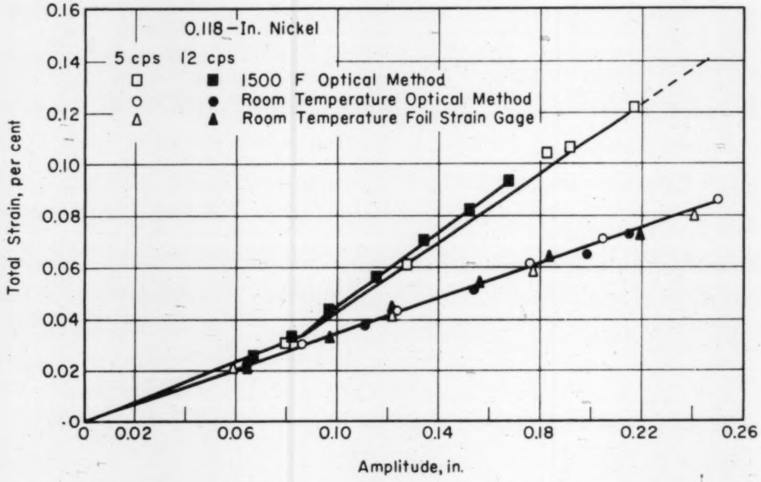


FIG. 4.—Strain Measurement Calibration Curves for 0.118 in. Thick Nickel Cycled at 5 and 12 cps.

section it probably holds for practical purposes. From Fig. 2,

$$r = \frac{g}{\phi} \qquad (2)$$

where g is the gage length, or distance between rods, and

$$\phi = \frac{1}{2}(\theta_1 - \theta_2) = \frac{1}{2}\left(\frac{l_1}{2b} - \frac{l_2}{2b}\right) = \frac{1}{4b}(l_1 - l_2) \qquad (3)$$

where b is the length of the optical lever and l is the length of the streak. These relationships hold if $l \ll b$. Finally,

$$\epsilon_0 = \frac{d}{4bg}(l_1 - l_2) \qquad (4)$$

Owing to the variation of specimen width over the gage length, the following correction is applied to convert average strain over this length to strain at the center of the reduced section:

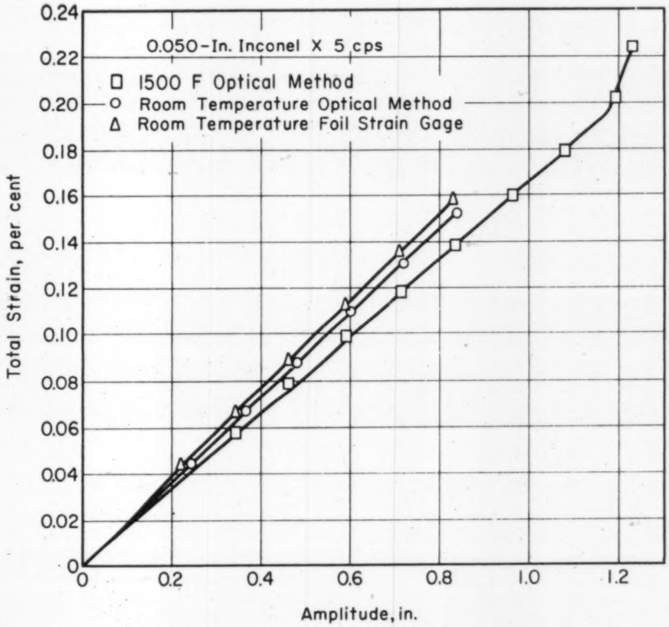


FIG. 5.—Strain Measurement Calibration Curves for 0.050 in. Thick Inconel X Cycled at 5 cps.

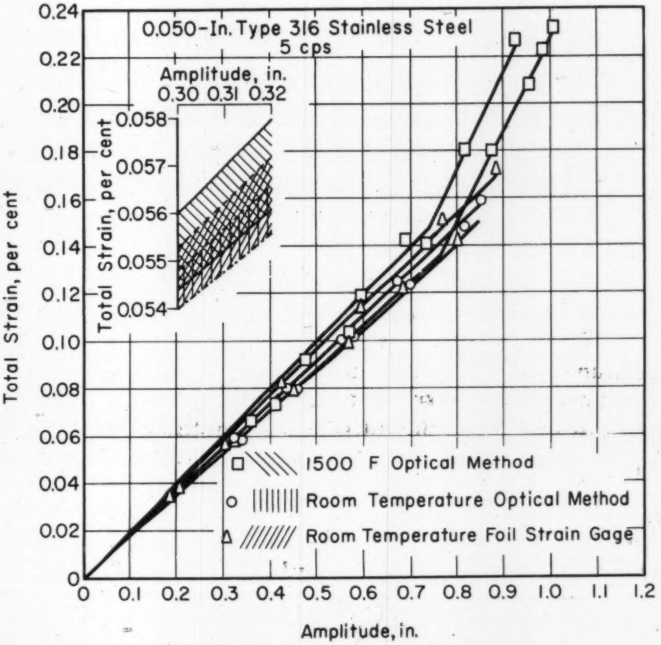


FIG. 6.—Strain Measurement Calibration Curves for 0.050 in. Thick Type 316 Stainless Steel Cycled at 5 cps.

Scatter bands for optical and foil strain gage methods are shown.

$$\epsilon = \epsilon_0 \frac{\bar{x}}{w} \qquad (5)$$

where:

ϵ_0 = uncorrected strain per half cycle, from strain gage or optical method,

ϵ = corrected strain per half cycle,

$\bar{x}$ = average width along gage length, and

w = minimum width of reduced section.

To establish the accuracy of the method, optical strain measurements at room temperature were compared to those made with a resistance gage before calibration runs at 1500 F on the same specimen.

Test Procedure:

Since the resonant frequency decreases with increased amplitude, the correct tuning weight to give the desired frequency was attached before sealing the vacuum chamber. The system was evacuated cold, heated to test temperature, and maintained there until the desired pressure was achieved, approximately 8 hr. The corresponding air tests were maintained at temperature for the same length of time. After reaching the desired vacuum the electrical contacts to the servomechanism were adjusted for an amplitude slightly smaller than the scheduled one, the resonant frequency was determined, and the amplitude then increased until the desired frequency was reached.

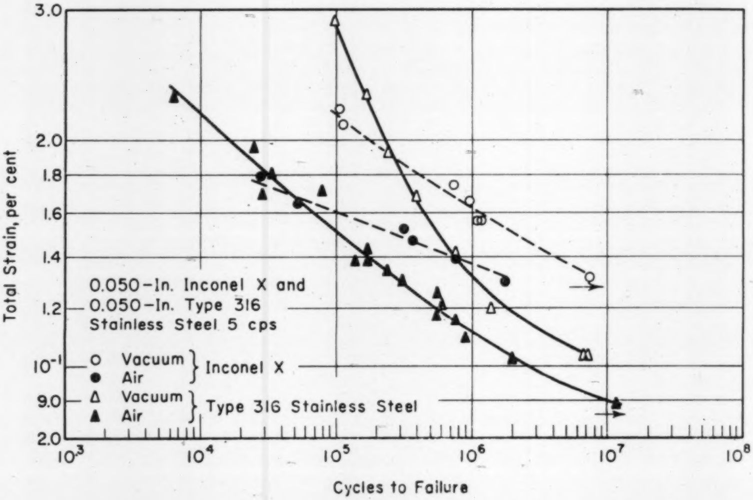


FIG. 7.—Vacuum and Air Fatigue Behavior of 0.050 in. Thick Inconel X and Type 316 Stainles Steel Cycled at 5 cps and 1500 F.

Average starting vacuum pressure: stainless steel, 1×10^{-5} mm of mercury; Inconel X, 2×10^{-6} mm of mercury.

For optimum control of vibration amplitude, the electromagnets were pulsed at a frequency slightly lower than the resonant frequency. For all except the highest amplitudes, where an excess of power was not available, the limit switches were adjusted to end the test when the magnet current had increased by 50 per cent. This setting, however, was not critical in affecting the fatigue life; as Wadsworth and Hutchings (3) have reported, the current remains fairly constant until just before failure.

TEST RESULTS

Strain Measurements:

Calibration curves for the three thicknesses of nickel are shown in Figs. 3 and 4. At room temperature the optical method substantially reproduced the strain-gage measurements. At 1500 F, however, the optical curves, probably because of the relatively low strength of nickel at high temperature, are considerably above the room-temperature values and display a change of slope evidently due to the transition from elastic to plastic bending. For Inconel X, in Fig. 5, the agreement for the two methods at room temperature is 3 per cent. Two hot runs, which differ by 3 per cent, are ap-

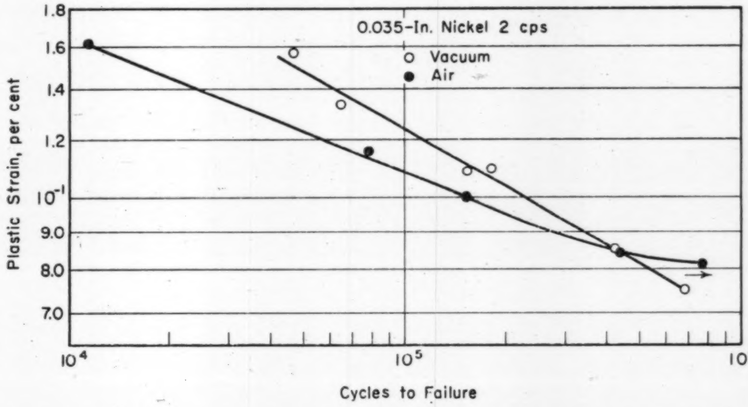


FIG. 8.—Vacuum and Air Fatigue Behavior of 0.035 in. Nickel Cycled at 2 cps and 1500 F.
Average starting vacuum pressure 5×10^{-6} mm of mercury.

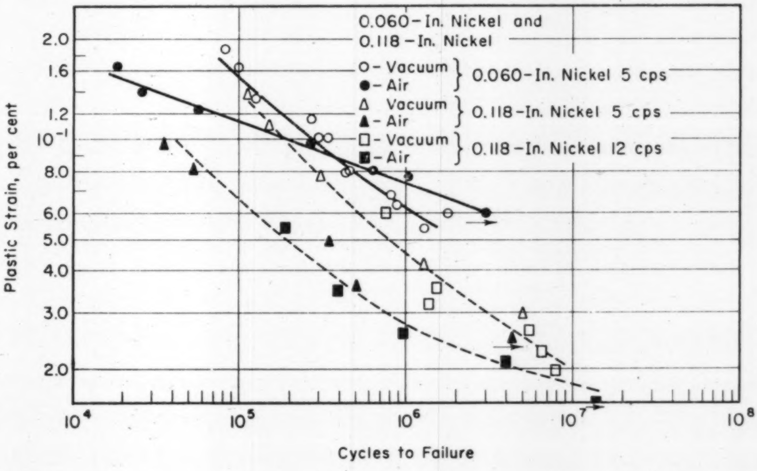


FIG. 9.—Effect of Section Size and Frequency on Vacuum and Air Fatigue Behavior of Nickel at 1500 F.

Average starting vacuum pressure: 0.060 in. thick nickel, 1×10^{-5} mm of mercury; 0.118 in. thick nickel, 2×10^{-6} mm of mercury.

proximately 5 per cent lower than the room-temperature curves. Continued precipitation hardening at 1500 F, following the final heat treatment at 1300 F, evidently stiffened the specimen and reduced the bending strain.

In order to establish the agreement between the two methods of strain measurement, a number of runs were made on the stainless steel. In Fig. 6 the extremes of each method are plotted both as full calibration curves and as an expanded section. Based on five runs at room temperature, the scatter band of 3 per cent for the optical method is contained within that for the strain-gage data of 5 per cent. Two runs at 1500 F differed by 5 per cent.

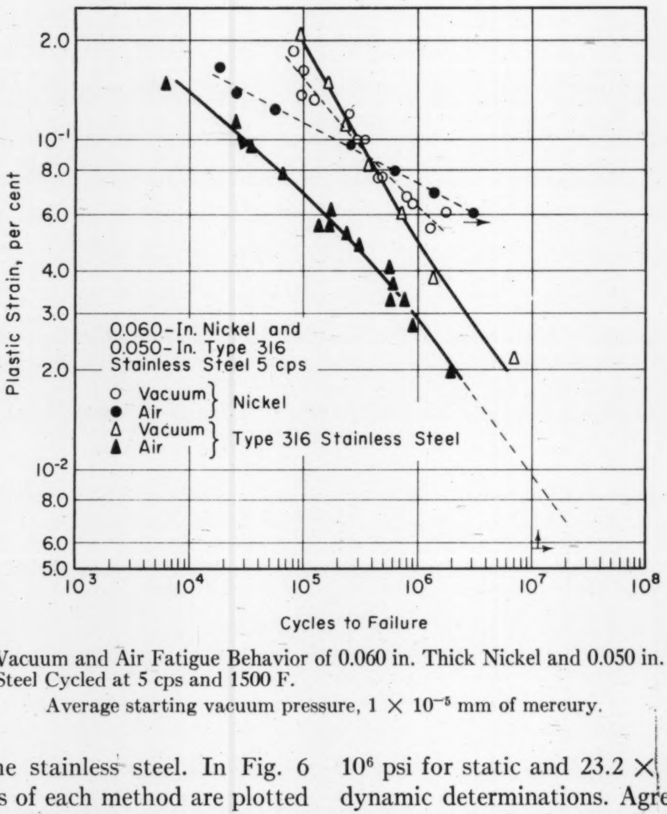


FIG. 10.—Vacuum and Air Fatigue Behavior of 0.060 in. Thick Nickel and 0.050 in. Thick Type 316 Stainless Steel Cycled at 5 cps and 1500 F.

Average starting vacuum pressure, 1×10^{-5} mm of mercury.

At room temperature the resistance strain gage was available as a standard, but at 1500 F an indirect method of comparison had to be used. A method described previously (7), which holds only for elastic strains, was used to calculate the stress for Inconel X. This calculated stress was then compared to the value obtained by converting measured strain, from the high-temperature optical method, to stress. Published values for the elastic modulus at 1500 F are 18.5×10^6 psi for static and 23.2×10^6 psi for dynamic determinations. Agreement for the two methods of stress determination is obtained if a value near the middle of this range, 20.4×10^6 psi, is assumed for the conversion.

Until an exhaustive evaluation of the accuracy and reproducibility of this method has been made, for purposes of reporting the results of this investigation, the data are considered reliable to 5 per cent.

Fatigue Study:

Following the procedure of Tavernelli and Coffin (9) the fatigue data, when possible, are plotted as log cycles to failure versus log per cent plastic strain per one-half cycle, which was computed by subtracting elastic strain, obtained from published values, from the total strain. Since the data on Inconel X are almost entirely in the elastic range, total strain was plotted instead of plastic strain.

The effect of environment on fatigue strength was, in general, similar for all three materials and appeared to conform to the competing-processes mechanism mentioned earlier. At high strains all the materials were stronger in vacuum, indicating that at short times, where oxidation is slight, the weakening mechanism by gas adsorption is controlling.

There is evidence, however, that oxidation strengthening was occurring simultaneously. In comparison to the fatigue life in vacuum, the air atmosphere reduced the fatigue life of stainless steel and Inconel X at high strains, in Fig. 7, by a large factor. The fatigue life of nickel, on the other hand, was reduced considerably less (see Figs. 8 and 9), possibly because it oxidized more readily. Also, in Fig. 10, while the fatigue behaviors of nickel and of stainless steel in vacuum are quite similar, in air there is a large difference; the nickel curve is much higher. For the same reason, as the strains are reduced, all the curves for vacuum and air data converge, although only those for the two thinner gages of nickel in Figs. 8 and 9 actually cross, indicating a reversal of the atmosphere effect.

Another indication of oxidation strengthening is the change in resonant frequency as the test progressed. In vacuum the resonant frequency always decreased—an indication of crack formation. In air, however, the tests at lowest strains on the two thicker gages of nickel showed increases in resonant frequencies of as much as 20 per cent as oxidation stiffened the specimen. No effect could be determined for the thinnest nickel because, owing to the large plastic strain, there was no resonant peak that could be used for the measurement. This progressive strengthening of nickel in air resulting in no failure after long times in test is an indication of a fatigue limit in air but not in vacuum.

Other indications of oxidation strengthening are to be found in the results of the study of section size and frequency effects in Fig. 9. The vacuum curves for the two gages of nickel are quite close, but in air the thinner specimens have considerably longer fatigue lives than the thicker ones. Similarly, in vacuum and at high strains in air there is no effect of frequency of vibration. But in air the increase of resonant frequency and prolonged fatigue life were observed at a higher strain at 5 cps than at 12 cps.

DISCUSSION

Previous investigations have shown that fatigue lives at room temperature can be considerably shorter in air than in vacuum. In the present work at 1500 F both weakening and strengthening effects of an oxidizing atmosphere have been found which may be explained by a proposed mechanism based on two processes acting in competition. Surface adsorption of gases facilitates crack propagation, while oxidation hardens and strengthens a metal.

Although a reversal in the atmosphere effect for the alloys is indicated only by extrapolation to very long times, there is the possibility, based on the temperature-dependence of the environmental effect in creep (5,6), that at higher temperatures even the alloys will have better fatigue strengths in air than in vacuum. Furthermore, investigations on alloys at higher temperature may demonstrate a fatigue limit in air but not in vacuum.

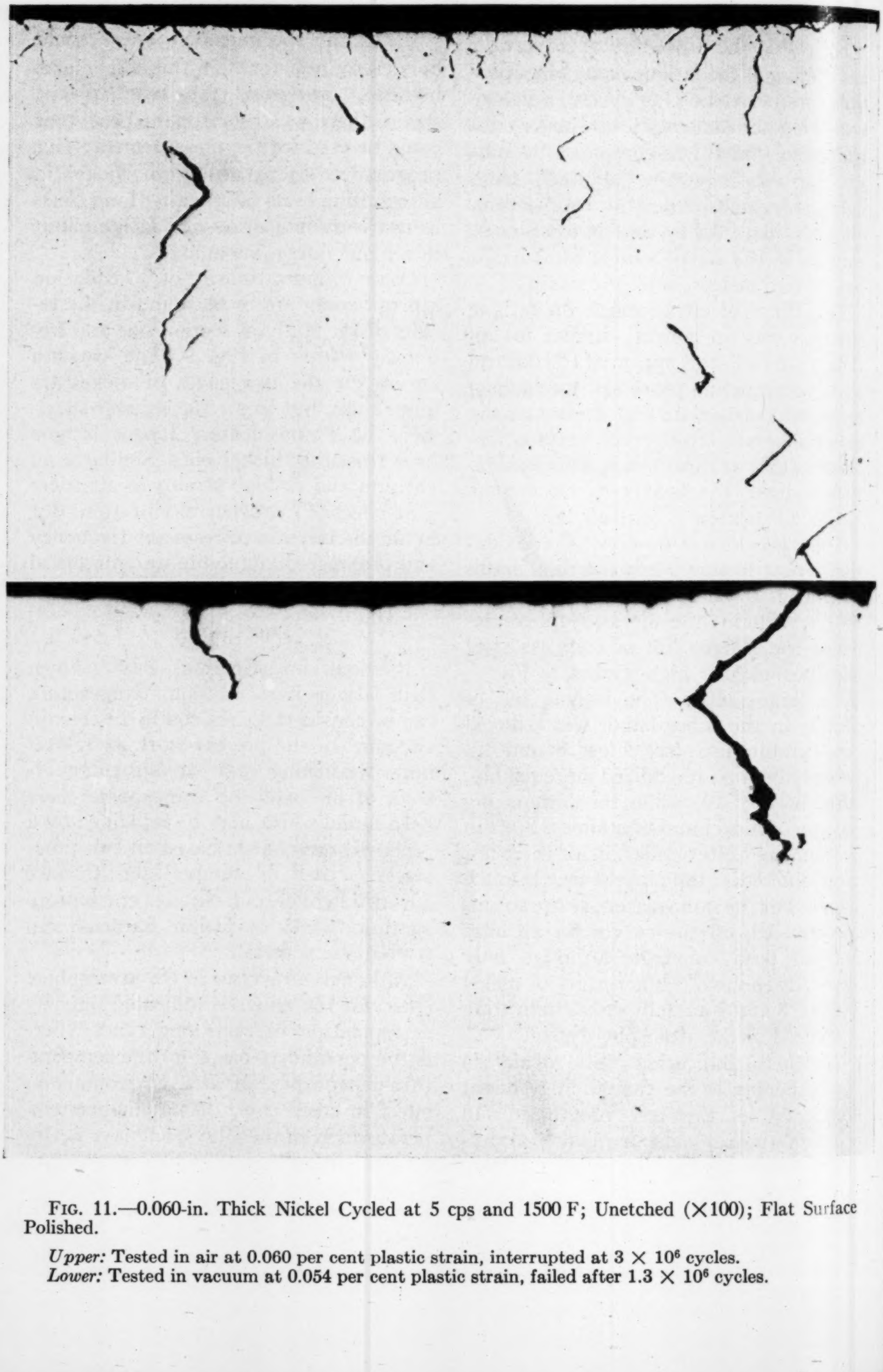


FIG. 11.—0.060-in. Thick Nickel Cycled at 5 cps and 1500 F; Unetched (×100); Flat Surface Polished.

Upper: Tested in air at 0.060 per cent plastic strain, interrupted at 3 × 10^6 cycles.
Lower: Tested in vacuum at 0.054 per cent plastic strain, failed after 1.3 × 10^6 cycles.

Since these questions are matters of concern to designers of high-temperature equipment, the possibility that fatigue strengths of commercial alloys may be higher in air than in a neutral environment should be examined with some care. First of all, the criterion for failure used in this work should be reappraised. In constant-amplitude machines the test is terminated by a drop in resonant frequency, which is taken to be an indication of the formation of cracks. But in Fig. 11, which is representative of crack structures in nickel, it can be seen that the interrupted specimen in air, which according to the above criterion did not fail, actually had more numerous cracks than the one that failed in vacuum. Evidently, filling of the cracks with an oxide layer, which can support a normal tensile stress (**10**), has prevented failure.

In the light of the observation that the supposedly stronger specimen actually has more cracks, it would appear that further work on the effect of atmosphere on crack formation and the properties of the resultant structure is in order. It is planned to interrupt tests in air and in vacuum to compare crack structure. Studies will also be made of the load-carrying ability of a metal which has been "strengthened" by oxidation.

SUMMARY AND CONCLUSIONS

Strain Measurements:

1. An optical technique for measuring bending strains in the plastic range at 1500 F has been developed for the purpose of reporting the fatigue results of this investigation.

2. Pending more exhaustive evaluation of the technique, strain measurement data at 1500 F are considered reliable to 5 per cent.

Fatigue Study:

1. In general, the fatigue test results at 1500 F were similar for the three materials investigated: nickel, type 316 stainless steel, and Inconel X. At high strains the fatigue lives are superior in vacuum, and with decreasing strain the vacuum and air curves converge.

2. The results of this investigation can be explained by a mechanism previously proposed to interpret similar findings in creep, which involves the simultaneous operation of two competing processes: The surface adsorption of gaseous impurities lowers surface energy and facilitates crack propagation, while oxidation provides a strengthening tendency.

3. That these processes operate simultaneously is evident when the environmental behavior of the nickel and the stainless steel are compared. At high strains the air atmosphere reduces the fatigue life by a factor of roughly 20, but the strength of nickel is reduced by only a factor of roughly 5 because it oxidizes more readily.

4. For the same reason the converging vacuum and air curves of the two thinner gages of nickel actually cross to produce a reversal in the environmental effect. A similar reversal may very well occur if commercial high-temperature alloys are tested at higher temperatures.

5. Further evidence of oxidation strengthening is the increase in resonant frequency of the two thicker nickel specimens when cycled at the lowest strain; this is indicative of stiffening.

6. A metallographic comparison of a nickel specimen tested in air and interrupted before failure with a specimen tested at nominally the same strain in a vacuum shows that more cracks exist in the supposedly stronger air-tested specimen than in the one tested to failure in vacuum. This suggests a reappraisal of the present criterion for fatigue failure in which the test is terminated by a drop in resonant frequency.

7. The results of a study of section size

and frequency effects are compatible with the proposed mechanism.

Acknowledgment:

The authors wish to thank A. C. Yingling for his help in construction of the equipment and conduct of the experimental program and A. R. Donaldson and D. H. Price for their performance of the metallographic work.

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DISCUSSION

MR. F. J. WALL.[1]—The curves representing data obtained in vacuum appear to slope downward whereas the curves for the data obtained in air appear to level off. Do the authors feel that the vacuum curves will level off to a definite endurance limit at a larger number of test cycles?

MR. G. J. DANEK (*author*).—No, I do not think there will be an endurance limit in vacuum. We did not get any indication of one during the longest fatigue test we were able to run in vacuum.

MR. CLARENCE R. SMITH.[2]—If there were sufficient vacuum, would it be possible that the lower endurance on the long-lived specimens might have been caused by cavitation rather than by annealing?

MR. DANEK.—Room-temperature experiments by Wadsworth and Hutchings have shown that we get no converging of fatigue curves since there is no oxidation, but rather a parallelism between the air and vacuum fatigue curves; this might help to answer your question.

MR. SMITH.—That is not what I had in mind. Some alloys, particularly magnesium, become unstable at very high vacuums so that a lower fatigue strength might be expected.

MR. DANEK.—No evidence of cavitation from evaporation of constituents was observed on the vacuum specimens tested.

MR. M. SEMCHYSHEN.[3]—The development of oxide in the air-tested specimens is obvious. It is also possible that oxide films could develop on the specimens tested in vacuum. Depending upon the partial pressure of oxygen in the "vacuum," the extent of oxide film and its composition may be different from that developed in an air atmosphere. Did the authors check for the existence of an oxide film and its composition on specimens tested in a vacuum?

MR. DANEK.—We got no visible oxide in the vacuum except for Inconel X. We have made no analysis of composition of oxide in any case.

MR. E. H. SCHUETTE.[4]—The size effect on the specimens tested in air is explained by stating that a greater percentage of the material was oxidized in the thinner specimens. Since the surfaces were presumably the same, this implies a subsurface effect of the oxidation, and it could therefore be taken as an indirect implication that failure initiation was subsurface. Were the specimens examined after testing for indications of subsurface initiation?

MR. DANEK.—No, the specimens were not examined for subsurface initiation.

MR. G. J. DANEK (*authors' closure*).—The possibility proposed by Mr. Smith that cavitation due to the vacuum environment may decrease strength has been brought up previously in connection with creep rupture studies at Naval Research Laboratory. In creep, materials were tested in helium and argon, to eliminate any effect of cavitation, as well as in vacuum. Rupture strengths in the inert gases were substantially the same as those resulting from vacuum-tested specimens.

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[2] Design Specialist, Convair, Division of General Dynamics Corp., San Diego, Calif.

[3] Director, Metallurgical Research, Climax Molybdenum Co., Detroit, Mich.

[4] Staff Engineer, Missiles and Space Division, Lockheed Aircraft Co., Sunnyvale, Calif.

In reference to the interesting discussion of Mr. Schuette concerning the reported size effect, the authors wish to submit a more detailed reply. One of the mechanisms by which oxidation can strengthen a metal is bulk oxidation which hardens and strengthens the bulk of the material. It is this process that is proposed to account for the reported size effect. As the fatigue test in air progresses, thinner specimens are stiffened by oxidation to a greater extent than thicker ones. Since these are constant amplitude tests, the plastic strain per half cycle decreases as the specimen stiffens, resulting in a decrease of crack propagation rate. There might not be a size effect, then, if plastic strain instead of amplitude were kept constant during the fatigue test.

ELASTOPLASTIC CYCLIC DEFORMATION AND LOW-CYCLE FATIGUE

BY S. V. SERENSEN[1] AND R. M. SHNEIDEROVITCH[2]

SYNOPSIS

Low-cycle fatigue fractures occur as a result of repeated elastoplastic deformation. In the case of nonuniform state of stress (for example, bending, torsion, stress concentration) the stress distribution changes from cycle to cycle. This paper is devoted to the investigation of stress-strain curves for metals under alternating loading and to the analysis of relative cyclic stress distribution. For this purpose, on the basis of experimental data an expression for stress-strain relation and plasticity function depending on the number of cycles is formulated.

When solving problems of alternating stress distribution for bars, plates, and shells according to the hypothesis of plane section the integral expression of plasticity function is proposed. The solution of this type of problem is given in terms of the solution of the same problem in the case of single loading. The conditions of low-cycle fatigue failure in the case of constant stress amplitude and constant strain amplitude loading are compared.

Elastoplastic cyclic deformation occurs under low-cycle fatigue loading. In general, this deformation results in a change in the stress amplitude of a machine part from cycle to cycle until rupture takes place.

At the present time, low-cycle fatigue testing is carried out either at a constant stress amplitude (**1,2**)[3] or at a constant strain amplitude (**3,4**). In both cases, for a quantitative analysis of failure condition, it is necessary to investigate the state of stress under elastoplastic cyclic deformation and the value of the rupture stress as a function of the number of cycles. In this connection, one should study cyclic stress-strain diagrams.

Such diagrams in this paper were obtained by testing tubular specimens in torsion. The wall thickness was 1 mm and the internal diameter was 20 mm.

Shearing deformation was measured by means of strain gages attached to a special device. Stresses were measured with strain gages attached to a torsion dynamometer. The stress-strain diagram was recorded by a mechanical-electronic recorder. Figure 1 illustrates a typical record. By means of limit contacts either the deformation amplitude or the stress amplitude was automatically held constant with any cycle nonsymmetry.

Figure 2 is a schematic stress-strain diagram of cyclic deformation plotted in dimensionless coordinates. The stresses are divided by the proportional limit,

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

σ_y, and the deformations are divided by the deformation, e_y, corresponding to the proportional limit. Such dimensionless quantities are indicated by placing a bar over the symbol. There are two systems of coordinates shown in Fig. 2: (1) the $\bar{\sigma}$-$\bar{e}$ system, with origin at point O, gives the total values of deformation; and (2) the $\bar{S}$-$\bar{\epsilon}$ system starting at point O', which refers to a given half cycle, k. The plastic component of deformation, $\bar{\epsilon}_p^{(k)}$, in the kth half cycle is equal to the hysteresis loop width, $\bar{\delta}^{(k)}$, when the maximum stress is reached.

To establish a general expression for the cyclic deformation curve, the yield strength, $\bar{S}_y$, on the kth half cycle must be known. As shown by experiments, $\bar{S}_y$ is approximately equal to 2.

Masing (5) established $\bar{S}_y$ to be 2 for the first half cycle. However, accurate measurement of $\bar{S}_y$ is difficult because of the nonlinearity of unloading, so $\bar{S}_y = 2$ is accepted throughout.

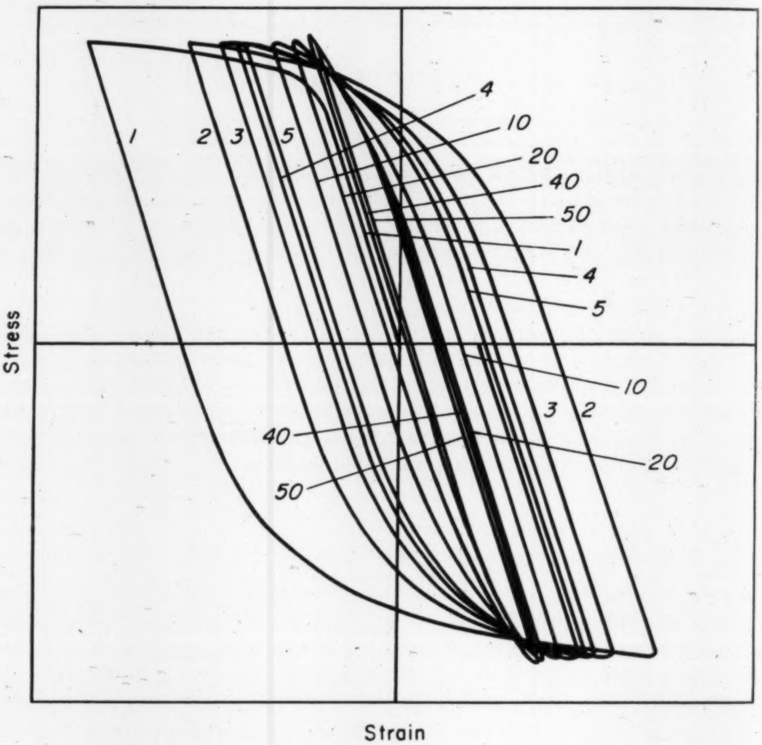


FIG. 1.—Record of Cyclic Deformation Diagram for an Aluminum Alloy. The numbers on the curves indicate the number of cycles.

Experimental cyclic stress-strain curves of some steels and light alloys for completely reversed stressing show the following relationship between the plastic components of deformation on the initial and kth half cycles (6,7):

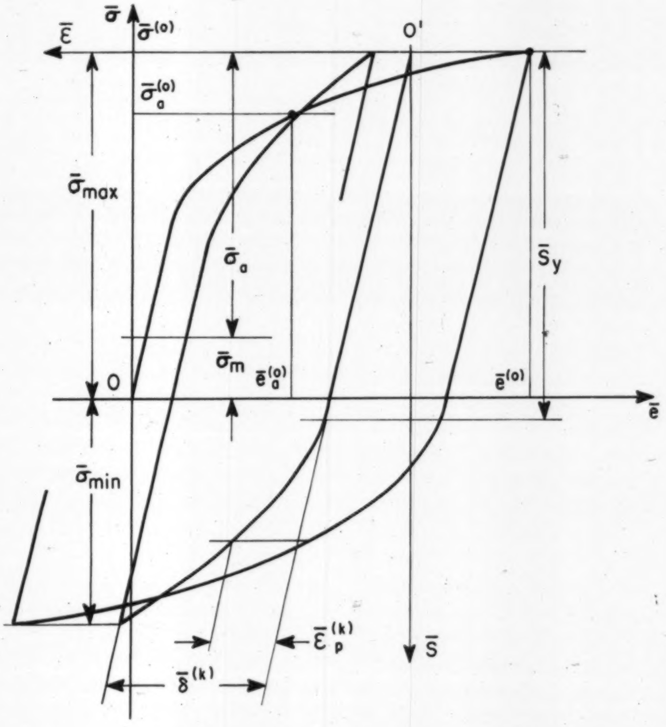
$$\bar{\epsilon}_p^{(k)} = \frac{A}{k^{\alpha}} (\bar{e}^{(0)} - 1)$$

Here $\bar{e}^{(0)}$ is the deformation on the zero half cycle and is a function of $\bar{\sigma}^{(0)}$. A and α are parameters of cyclic deformation.

The curve of cyclic deformation for nonsymmetrical loading is generally determined by the stress amplitude, resulting in a similar expression:

$$\bar{\epsilon}_p{}^{(k)} = \frac{A}{k^{\alpha}} (\bar{e}_a{}^{(0)} - 1)$$

where $\bar{e}_a{}^{(0)}$ is the deformation on the zero half cycle at a stress equal to the stress amplitude $\bar{\sigma}_a$.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O'
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$\bar{\sigma}_m$
$\bar{e}_a^{(0)}$
$\bar{e}^{(0)}$
O
$\bar{e}$
$\bar{\sigma}_{min}$
$\bar{\varepsilon}_p^{(k)}$
$\bar{S}$
$\bar{\delta}^{(k)}$

FIG. 2.—Schematic Stress-Strain Diagram of Cyclic Deformation.

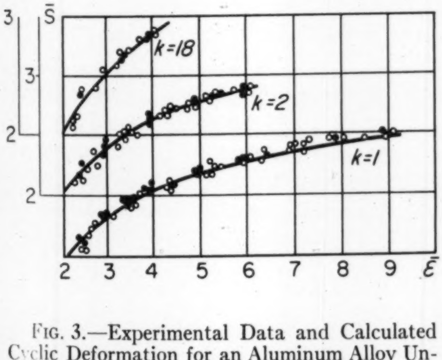

FIG. 3.—Experimental Data and Calculated Cyclic Deformation for an Aluminum Alloy Under Constant-Stress-Amplitude Loading.

An equation for the stress-strain diagrams can then be formulated as follows:

$$\bar{\epsilon}^{(k)} = \frac{A}{k^{\alpha}} \left[f\left(\frac{\bar{S}}{2}\right) - 1 \right] + \bar{S}$$

In this way the cyclic stress-strain curve can be expressed in terms of the initial stress-strain curve. Stress-strain curves calculated by this equation for an aluminum alloy for a symmetrical cycle (open circles) and a nonsymmetrical cycle (solid circles) are compared in Fig. 3.

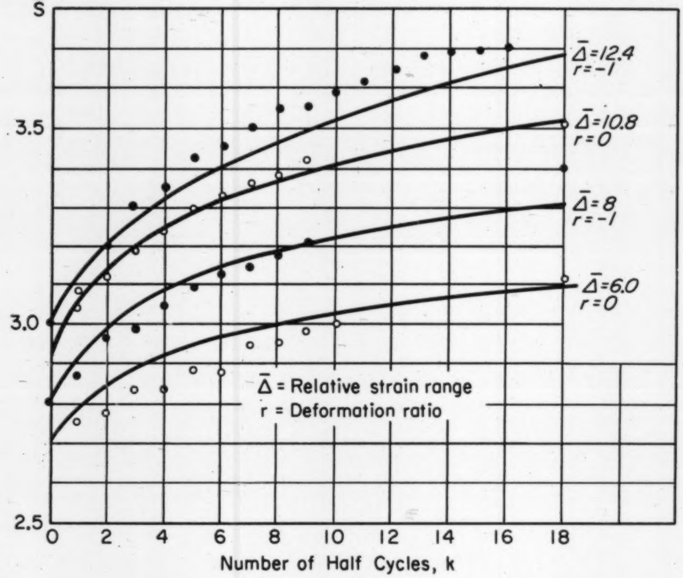

FIG. 4.—Experimental Data and Theoretical Curves Under Constant-Strain-Amplitude Loading.

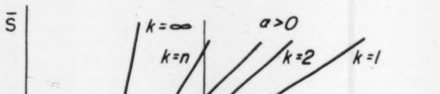

FIG. 5.—Combined Cyclic Deformation Curves for Various Numbers of Half Cycles.

The cyclic stress-strain curves mentioned above have been established from experimental data obtained under constant-stress-amplitude loading. The stress-strain curves for constant-strain-amplitude loading with a relative strain range $\bar{\Delta}$ may be described by the same expressions (7) if the deformation is taken to be constant:

$$\bar{\epsilon}^{(k)} = \frac{A}{k^{\alpha}}\left[f\left(\frac{\bar{S}}{2}\right) - 1\right] + \bar{S} = \bar{\Delta}$$

Comparison of theoretical and experimental curves of stress versus k under constant-strain-amplitude loading are plotted in Fig. 4 for different values of $\bar{\Delta}$ and r, the deformation ratio (minimum strain divided by maximum strain).

The parameter, α, of cyclic deformation curves determines their change with number of cycles. In Fig. 5 are plotted schematically the combined curves of cyclic stress-strain curves for different numbers of half cycles when $\alpha > 0$ and

$\alpha < 0$. Also shown are lines for constant-stress and constant-strain cycling.

Strengthening occurs when $\alpha > 0$. Under constant-stress-amplitude loading the width of a hysteresis loop decreases from cycle to cycle, whereas the stress increases under constant-strain-amplitude loading.

Softening occurs when $\alpha < 0$. The width of the loop increases for stress cycling and the stress decreases for strain cycling.

The value $\alpha > 0$ is typical of aluminum alloys; $\alpha < 0$ for high-strength steels and $\alpha \sim 0$ for carbon steels with a yield point.

Stress-strain curves under cyclic loading can be made linear if the static stress-strain curve is approximated by two straight lines having a slope E in the elastic region and a slope E_T in the work-hardening region. For the zero (initial) half cycle,

$$\bar{\sigma}_i^{(0)} = (1 - \bar{G}_T) + \bar{G}_T \bar{e}_i^{(0)} \quad (\bar{e}_i > 1)$$

where $\bar{G}_T = \dfrac{E_T}{E}$, the modulus of strengthening, and $\bar{\sigma}_i^{(0)}$ and $\bar{e}_i^{(0)}$ are the stresses and strains for the initial half cycle. The expression for the cyclic stress-strain curve in this case is:

$$\bar{S}_i^{(k)} = 2[1 - g(k)] + g(k)\bar{\epsilon}_i^{(k)}$$

where $g(k) = \dfrac{1}{1 + A/2\bar{G}_T k^\alpha}$, the modulus of cyclic strengthening, and $\bar{S}_i$ and $\bar{\epsilon}_i$ are the values of stresses and strains on the kth half cycle. Using the coordinates $S_i^* = \dfrac{\bar{S}_i}{\bar{S}_y}$ and $\epsilon_i^* = \dfrac{\epsilon_i^*}{\epsilon_y}$, where, as the first approximation, $\bar{S}_y = 2$ and $\bar{\epsilon}_y = 2$, in the case of cyclic deformation an equation similar to that for deformation under single loading is obtained:

$$S_i^{*(k)} = 1 - g(k) + g(k)\epsilon_i^{*(k)} \quad (\epsilon_i^* > 1)$$

$$S_i^{*(k)} = \epsilon_i^{*(k)} \quad (\epsilon_i^* < 1)$$

The stress-strain ratio or plasticity function, ϕ, under cyclic and static deformation is similar:

$$\phi^{(k)} = \frac{S_i^{*(k)}}{\epsilon_i^{*(k)}} = \frac{1 - g(k)}{\epsilon_i^{*(k)}} + g(k)$$

$$\phi^{(0)} = \frac{\bar{\sigma}_i^{(0)}}{\bar{e}_i^{(0)}} = \frac{1 - \bar{G}_T}{\bar{e}_i^{(0)}} + \bar{G}_T$$

The plasticity function can easily be determined when the single loading plasticity for the same amount of deformation is known:

$$\phi^{(k)} = 1 - \frac{1 - g(k)}{1 - \bar{G}_T} (1 - \phi^{(0)})$$

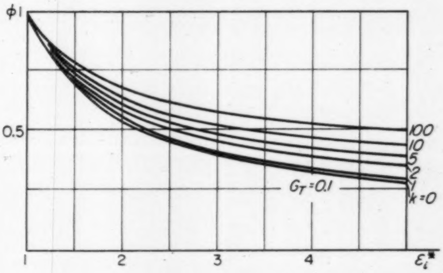

FIG. 6.—Curves of the Plasticity Functions for Various Numbers of Half Cycles.

In Fig. 6 curves are plotted for the plasticity function at different numbers of half cycles when $\bar{G}_T = 0.1$, $A = 1.5$, and $\alpha = 0.5$.

When solving problems posed by plastic deformation of bars, plates, and shells using the hypothesis that plane sections remain plane, integral expressions for the plasticity function are used (**8**):

$$\phi_t = k_t \int \phi \, d\eta$$

and

$$\phi_b = k_b \int_\eta \phi\eta^2 \, d\eta$$

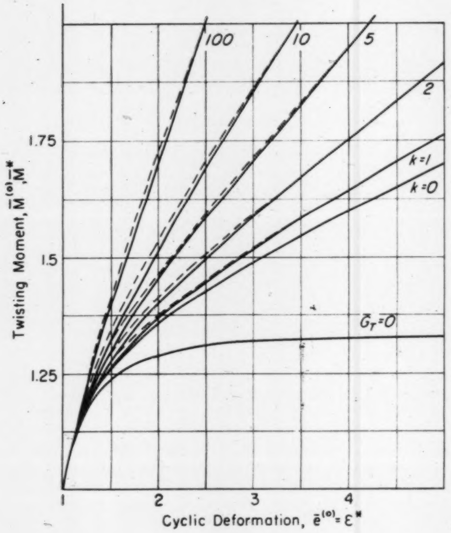

FIG. 7.—Effect of Cyclic Deformation on Torque.

where η is the relative height of the cross-section, and k_t and k_b are the coefficients for the cross-sectional form. As cited above, for the function ξ, $\phi^{(k)}$ can be expressed in terms of the function $\phi^{(0)}$ for static loading:

$$\phi^{(k)} = 1 - \frac{1 - g(k)}{1 - \bar{G}_T}\,(1 - \phi^{(0)})$$

On the basis of this integral expression for the plasticity function, it is possible to express the force for some k half cycle in terms of the force under static loading for the same deformation:

$$Q^{*(k)} = \frac{1 - g(k)}{1 - \bar{G}_T}\,\bar{Q}^{(0)} + \frac{g(k) - \bar{G}_T}{1 - \bar{G}_T}\,\bar{e}^{(0)}$$

where Q^* is the load acting in cross-section and $\bar{e}^0 = \epsilon^*$, where ϵ^* is the deformation corresponding to this load. It is necessary to take into consideration that $\epsilon^* = Q^*_{\text{elast}}$.

The problem of stress distribution in the case of cyclic elastoplastic deformation can be solved, using either ϕ, the integral expression of the plasticity function mentioned above, or by solving as in static loading. This is shown in Fig. 7 for the case of torsion of a circular bar, which shows the twisting moment as a function of deformation at $\bar{G}_T = 0.1$, $A = 1.5$, and $\alpha = 0.5$ for different numbers k of half cycles. From these curves

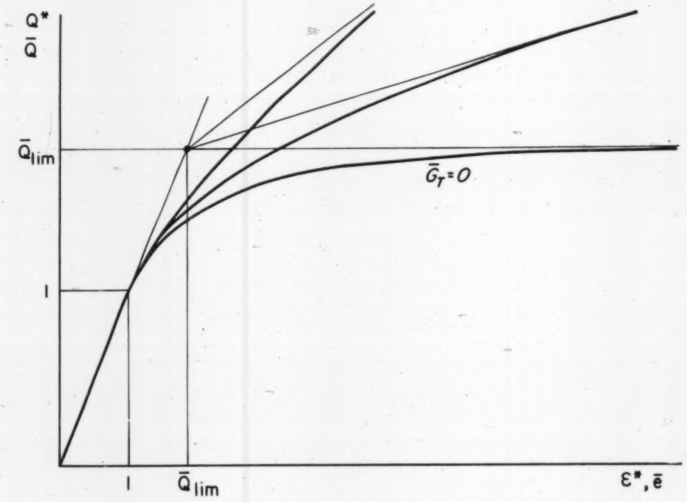

FIG. 8.—Dependence of Load on Deformation.

it is possible to calculate the values of deformation ϵ^* in a given half cycle for a given amplitude of acting force. After determining the deformation it is possible to obtain the value of stress. This way of solving cyclic elastoplastic problems is somewhat involved, as it is necessary to calculate the deformation in each half cycle. An approximate expression of the function may be stated, assuming linear strengthening and taking into account the asymptotic properties of the curves showing the relation between the load Q and the deformation ϵ.

In the case of plasticity without strengthening $\bar{G}_T = 0$, and for large deformations, the limiting load $\bar{Q}_{\text{lim}}$ corresponding to the fully plastic state is reached. When the modulus of strengthening $\bar{G}_T$ is other than zero, the value of the load corresponding to the deformation $\bar{e}$ is (Fig. 8):

$$\bar{Q} = \bar{Q}_{\text{lim}}\,(1 - \bar{G}_T) + \bar{e}\bar{G}_T$$

When dealing with cyclic deformation on the kth half cycle, the value of this load is

$$Q^{*(k)} = \bar{Q}_{\text{lim}}\,[1 - g(k)] + \epsilon^{*(k)}g(k)$$

According to this the deformation is

$$\epsilon^{*(k)} = \frac{Q^*}{g(k)} - \bar{Q}_{\text{lim}}\,\frac{1 - g(k)}{g(k)}$$

$$= Q^* + (Q^* - \bar{Q}_{\text{lim}})\,\frac{A}{2\bar{G}_T k^{\alpha}}$$

Taking into account the similarity of expressions for the limiting loads $\bar{Q}_{\text{lim}}$ and the stress-strain curves and using geometric considerations, it is possible to establish the following relations:

For loads:

$$Q^{*(k)} - \bar{Q}_{\text{lim}} = \frac{\bar{e}^{(0)} - \bar{Q}^{(0)}}{1 - \bar{G}_T}\,\frac{A}{2k^{\alpha}}$$

For deformations:

$$\epsilon^{*(k)} = Q^* + \frac{\bar{e}^{(0)} - \bar{Q}^{(0)}}{1 - \bar{G}_T}\,\frac{A}{2k^{\alpha}}$$

Here $\bar{e}^{(0)}$ and $\bar{Q}^{(0)}$ are the deformation and the force corresponding to this deformation for the initial (zero) half cycle.

This expression gives precise values for large deformations and can be used as an approximation for any value of $\bar{e}^{(0)}$. Similarly, the deformation, $\epsilon^{*(k)}$, in the kth half cycle under nonsymmetrical cyclic loading is as follows:

$$\epsilon^{*(k)} = Q_a^* + \frac{\bar{e}_a^{(0)} - \bar{Q}_a^{(0)}}{1 - \bar{G}_T}\,\frac{A}{2k^{\alpha}}$$

For the problem of cyclic torsion of a circular bar the approximate values of $Q^{*(k)}$ can be calculated from the expression given above. The corresponding results are plotted in Fig. 7 by dashed lines.

The above equations give the cross-sectional deformation corresponding to the maximum elastic deformation for the given type of loading, and in this case

$$Q_a^* = \epsilon_{a\,\text{elast}}^{*\text{max}}$$

and

$$\bar{Q}_a^{(0)} = \bar{e}_{a\,\text{elast}}$$

Other cross-sectional points may be represented by a more general expression:

$$\epsilon^{*(k)} = \overset{*}{\epsilon}_{a\,\text{elast}} + \frac{\bar{e}_a^{(0)} - \bar{e}_{a\,\text{elast}}}{1 - \bar{G}_T}\,\frac{A}{2k^{\alpha}}$$

Here $\epsilon^*_{a\,\text{elast}} = \bar{e}_{a\,\text{elast}}$ is the amplitude of deformation in the given cross-sectional point in the case of elastic deformation under loading $\bar{Q}_a$; $\bar{e}_a^{(0)}$ is the deformation in this cross-sectional point under single loading $\bar{Q}_a$.

A similar expression can be obtained for the elastoplastic displacement:

$$\Delta^{*(k)} = \overset{*}{\Delta}_{a\,\text{elast}} + \frac{\Delta_a^{(0)} - \bar{\Delta}_{a\,\text{elast}}}{1 - \bar{G}_T}\,\frac{A}{2k^{\alpha}}$$

The expressions for stresses S^*, deformations ϵ^*, and displacements mentioned above are for their values during one cycle. In general, values of accumulated deformations after the *k*th half cycle are taken to be equal to:

$$\bar e^{(k)} = \bar e^{(0)} + 2\sum_{k=1}^{k} \epsilon^{*(k)}(-1)^k$$

$$\bar\sigma^{(k)} = \bar\sigma^{(0)} + 2\sum_{k=1}^{k} S^{*(k)}(-1)^k$$

$$\bar\Delta^{(k)} = \bar\Delta^{(0)} + 2\sum_{k=1}^{k} \Delta^{*(k)}(-1)^k$$

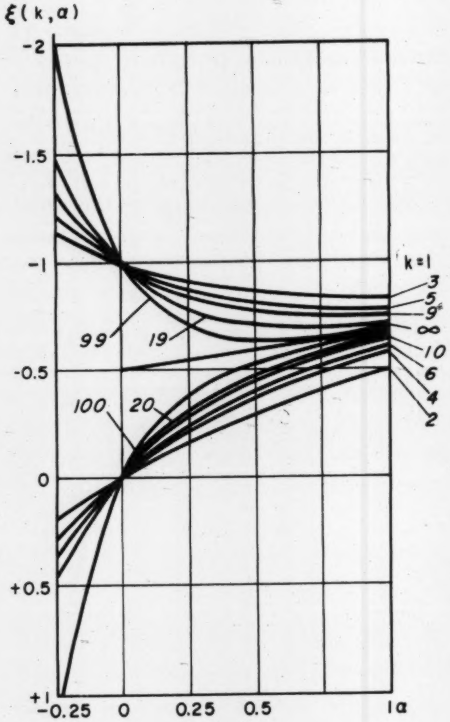

FIG. 9.—Illustrating the Function $\xi(k,\alpha) = \sum_{k=1}^{k} \frac{(-1)^k}{k^\alpha}$.

Here $\bar e^{(k)}$ = the components of deformation $\bar e_{xx}$, $\bar e_{yy}$, and so on, and $\bar\sigma^{(k)}$ = the components of stress $\bar\sigma_{xx}$, $\bar\sigma_{yy}$, and so on.

Deformation in nonsymmetrical cyclic loading with constant stress amplitude is equal to

$$\bar e^{(k)} = (\bar e^{(0)} - \bar e_a^{(0)}) + (\bar e_a^{(0)} - \bar e_{a\,\text{elast}}) \cdot \left[1 + \frac{A}{1-\bar G_T}\,\xi(k,\alpha)\right] + (-1)^k \bar e_{a\,\text{elast}}$$

The function $\xi(k, \alpha) = \sum_{k=1}^{k} \frac{(-1)^k}{k^\alpha}$ can be calculated beforehand. In Fig. 9 this function is plotted as a function of the parameter α for different numbers of half cycles. The sum of the series

$$\sum_{k=1}^{\infty} \frac{(-1)^k}{k^\alpha} = \xi(\alpha) \quad \text{at} \quad \alpha > 0$$

converges to the function $\xi(\alpha)$ connected with Riemann's zeta function $\zeta(\alpha)$ by the relation

$$\xi(\alpha) = -(1 - 2^{1-\alpha})\zeta(\alpha)$$

This function can be approximately expressed by the following equation:

$$\xi(\alpha) = (0.5 - \ln 2)\alpha - 0.5$$

Similarly, displacements in some *k*th half cycles are equal to:

$$\bar\Delta^{(k)} = (\bar\Delta^{(0)} - \bar\Delta_a^{(0)}) + (\bar\Delta_a^{(0)} - \bar\Delta_{a\,\text{elast}}) \cdot \left[1 + \frac{A}{1-\bar G_T}\,\xi(k,\alpha)\right] + (-1)^k \bar\Delta_{a\,\text{elast}}$$

In these expressions, $\bar e^{(0)}$ and $\bar\Delta^{(0)}$ are the deformations and displacements, respectively, in the initial half cycle at the maximum loading.

After certain *k*th transformations, the value of stresses after the *k*th half cycle in the nonsymmetrical cycle under constant-stress-amplitude loading can be expressed by the following equation:

$$\bar\sigma^{(k)} = (\bar\sigma^{(0)} - \bar\sigma_a^{(0)}) + (\bar\sigma_a^{(0)} - \bar\sigma_{a\,\text{elast}}) \cdot \left[1 + \frac{2a}{1-\bar G_T}\,\eta(k,\alpha,a)\right] + (-1)^k \bar\sigma_{a\,\text{elast}}$$

where:

$\bar\sigma^{(0)}$ = the component of stress arising in the initial (zero) half cycle at the maximum load $\bar Q_{\max}$,

$\bar\sigma_a^{(0)}$ = the component of stress in the

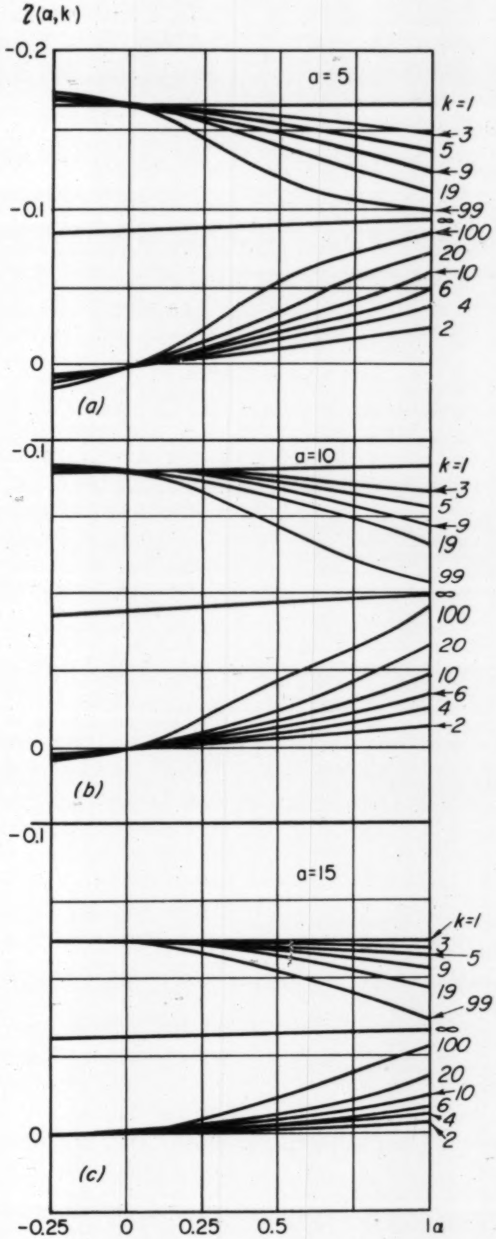

FIG. 10.—Illustrating the Function $\eta(k,\alpha,a) = \sum_{k=1}^{k} \frac{(-1)^k}{a + k^\alpha}$, with: (*a*) $a = 5$, (*b*) $a = 10$, and (*c*) $a = 15$.

initial half cycle under the action of the load $\bar{Q}_a$, and

$\bar{\sigma}_{a\ \mathrm{elast}}$ = the component of stress if the deformation under the load $\bar{Q}_a$ were totally elastic.

The function $\eta(k,\alpha,a) = \sum_{k=1}^{k} \frac{(-1)^k}{a+k^\alpha}$ can be calculated beforehand for the assigned constant values α and $a = \frac{A}{2\bar{G}_T}$ and the number of half cycles. The curves of η for $a = 5$, 10, and 15 are plotted in Fig. 10. When $\alpha > 0$, the sum of the series $\sum_{k=1}^{\infty} \frac{(-1)^k}{a+k^\alpha}$ converges to the function $\eta(a,\alpha)$, and the values of η can be approximately calculated from the equation (for the whole values of a):

$$\eta(a,\alpha) = \left(\frac{0.5}{a+1} - |\ln 2 - \xi(a, 1)|\right)\alpha - \frac{0.5}{a+1}$$

where the function ξ (α, 1) can be determined from Fig. 10 for $k = a$ and $\alpha = 1$.

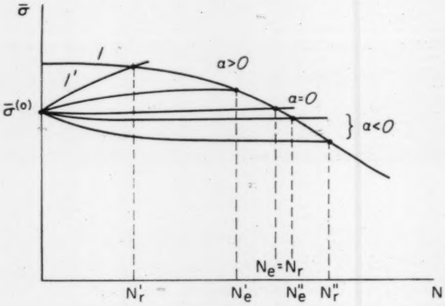

FIG. 11.—Intersections of Fatigue Curve and Stress Curves in a Machine Part.

The above expressions of ξ (k, α) and $\eta(k,\alpha,a)$ make it possible to determine the deformations $\bar{e}^{(k)}$ and stresses $\bar{\sigma}^{(k)}$ after the kth half cycle as well as the residual values after unloading in any given half cycle. These equations are based on data obtained by solving the problems of elastoplastic deformation under single loading.

APPLICATIONS

The above approach enables us to investigate the change in stress from cycle to cycle when testing specimens and machine parts. Under constant loading amplitude the situation is somewhere between stress cycling and strain cycling (Fig. 5). When $\alpha > 0$ the stresses in the machine part increase from cycle to cycle, but somewhat more slowly than in the case of constant-strain-amplitude loading. When $\alpha < 0$ the stresses decrease, also somewhat more slowly than under constant-strain-amplitude loading.

The low-cycle fatigue curve obtained under constant-stress-amplitude loading can be used for estimating the strength of a machine part. Plotting the stress acting in the machine part as a function of cycles on the fatigue curve enables us to estimate the number of cycles and the load at the time fatigue rupture occurs (Fig. 11, curves *1* and *1'*). In this case it is assumed that the fatigue curve is not influenced by any continuous change of stress amplitude. The above results enable us to compare data under both constant stress and constant strain amplitudes.

Figure 11 illustrates the relative scheme. When testing a machine part under constant load amplitude, rupture (initiation of cracking) takes place when the number of cycles is equal to N'_e. When testing under constant strain amplitude when $\alpha > 0$, more rapid increase in stress from cycle to cycle occurs, and fatigue cracking begins earlier, when the number of cycles is $N'_r < N'_e$. If $\alpha < 0$, when testing a machine part under constant stress amplitude a decrease in stress from cycle to cycle takes place, and fatigue cracking begins when the number of cycles is equal to $N''_e > N'_e$. Under constant-strain-amplitude loading, the number of cycles necessary for fatigue rupture will be still more ($N''_r > N'_r$). When the hysteresis loop is stable ($\alpha = 0$), the change of stress under cyclic loading does not take place. Conditions of testing a machine part under constant stress and constant strain amplitude, in this case, remain the same.

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FRACTOGRAPHIC ANALYSIS OF AISI 4340 STEEL BY OPTICAL AND ELECTRON MICROSCOPY*

By N. A. Tiner[1]

Synopsis

A study is made of the modes of fracture for AISI 4340 steel. The fracture surfaces are examined by electron microscopy. The discontinuous nature of fracture propagation is analyzed and two methods of observations are developed for determining the direction of propagation: (1) the observation of the concavity of the elongated domains on the shear fracture surface by electron microscopy, and (2) examination of the secondary crack orientation in the transverse fracture profile by optical microscopy.

It is well known that sheet steel shows two modes of fracture under the influence of an external tensile load: oblique shear and transverse fracture. The oblique shear occurs along the slip planes within individual crystals, and the gross plane of separation on a microscopic scale is inclined at about 45 deg to the direction of the tensile load, being the plane of maximum shear stress. The transverse fracture follows the cubic cleavage planes within individual crystals, and the gross fracture plane is normal to the direction of the tensile load (**1**).[2]

The transverse mode of fracture is in certain cases restricted by the development of shear lips. The portion of the thickness of a sheet occupied by these shear lips is usually referred to as the per cent shear. It is influenced to a large extent by the testing temperature, thickness of the sheet, composition, and heat treatment (**2, 3**).

The purpose of this report is to examine in detail these fracture appearances in AISI 4340 steel and to develop a method for determining the direction of crack propagation. A clear understanding of this problem is of great importance in the production of missile motor casings.

Shear Fracture

A shear fracture can be obtained by a double slant-notch shear specimen failed in axial tension. Figure 1 shows such a specimen and its mode of fracture for 4340 steel. The central area between the slots is under essentially a pure shear stress (**4, 5**). The electron micrographs of the replicas taken from the fracture surface in this area are included in the figure. It will be noted that the fracture exhibits parabola-like lines or elongated domains with smooth surfaces and a number of more or less extended flat zones. If one looks on the two opposite faces of the fracture, it is observed that the concavity of the domains are turned in opposite directions on the opposite faces, for each face viewed from above the concavity is oriented toward the direction of the relative displacement of the other half of the specimen.

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* Presented at the Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

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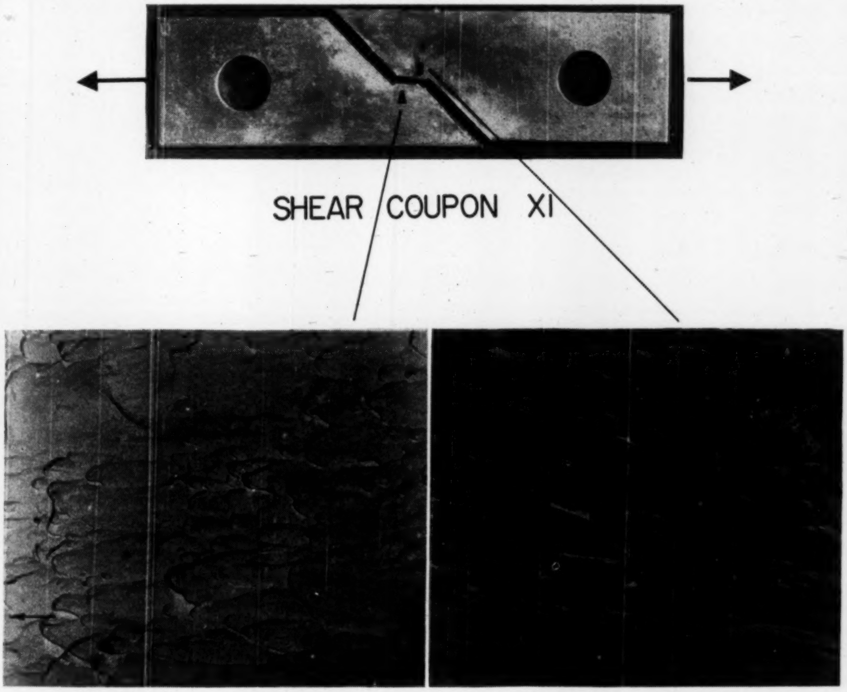
[2] The boldface numbers in parentheses refer to the list of references appended to this paper.



Surface A (×26,000). Surface B (×26,000).

FIG. 1.—Fracture of a Double Slant-Notch Shear Coupon of 4340 Steel.

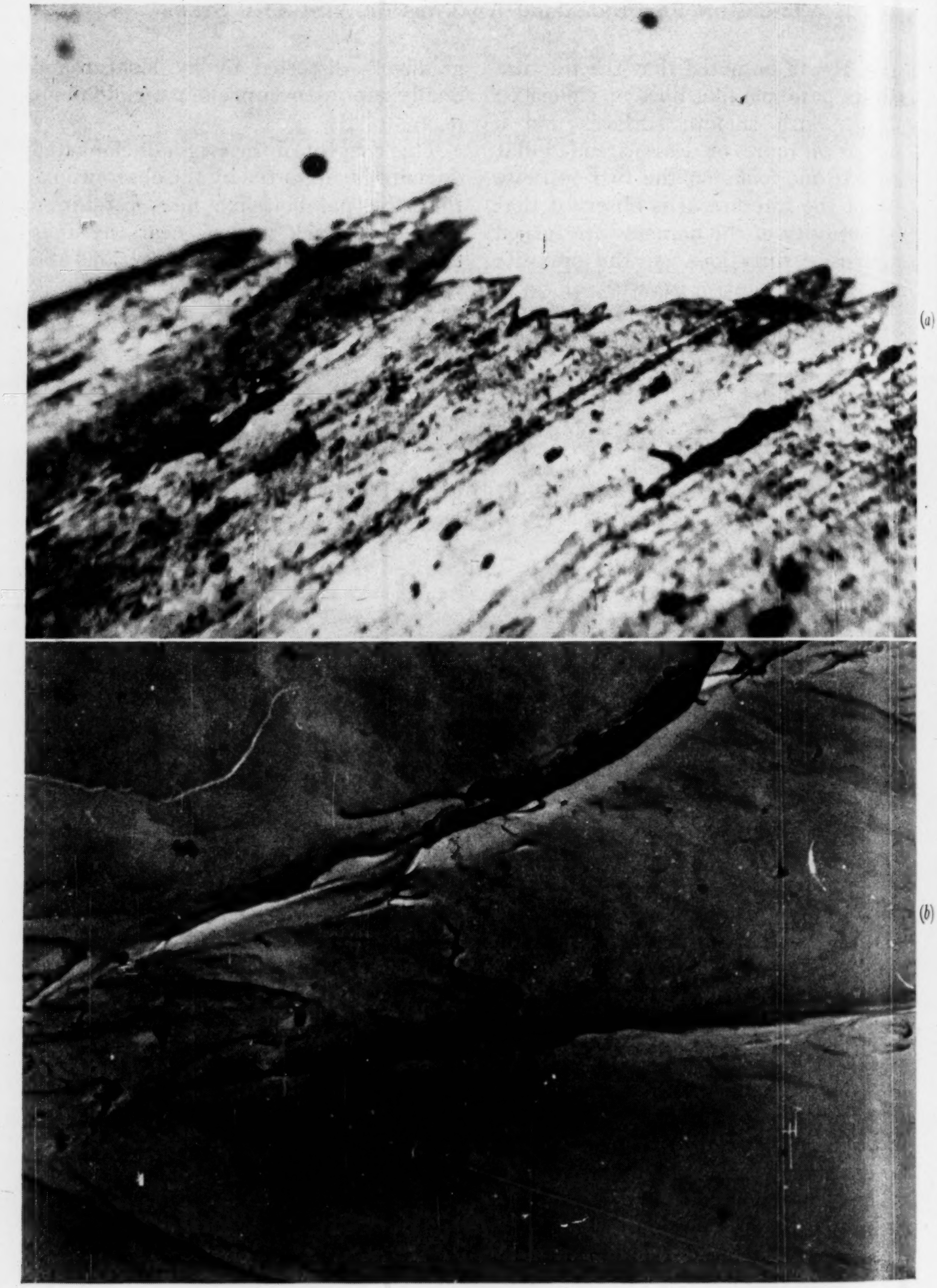
Electron micrographs are two-stage chromium shadowed carbon replicas of the fracture surfaces as indicated.

These parabola-like lines owe their visibility to the chromium shadow and excess carbon deposit resulting from the existence of sharp changes in level. They would be expected if microcracks are produced in advance of the main rupture front and then the microcrack is progressively distorted by local shear and finally rupture occurs along a glide plane (**6, 7**).

This concept of the origin of elongated domains is supported by the observations that the parabola-like lines contain a dent or a crack nucleus near the apex formed by the pileup of dislocations and that the interior is relatively smooth. Furthermore, the domains of an Armco iron specimen with relatively coarse grains (Fig. 2) are considerably larger than those of 4340 steel with small (martensite) crystals (Fig. 1).

TRANSVERSE FRACTURE

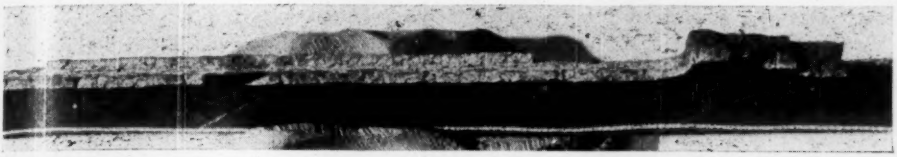
A transverse fracture surface may exhibit chevron pattern, hackle marks, parallel ridges, or a fibrous structure. A



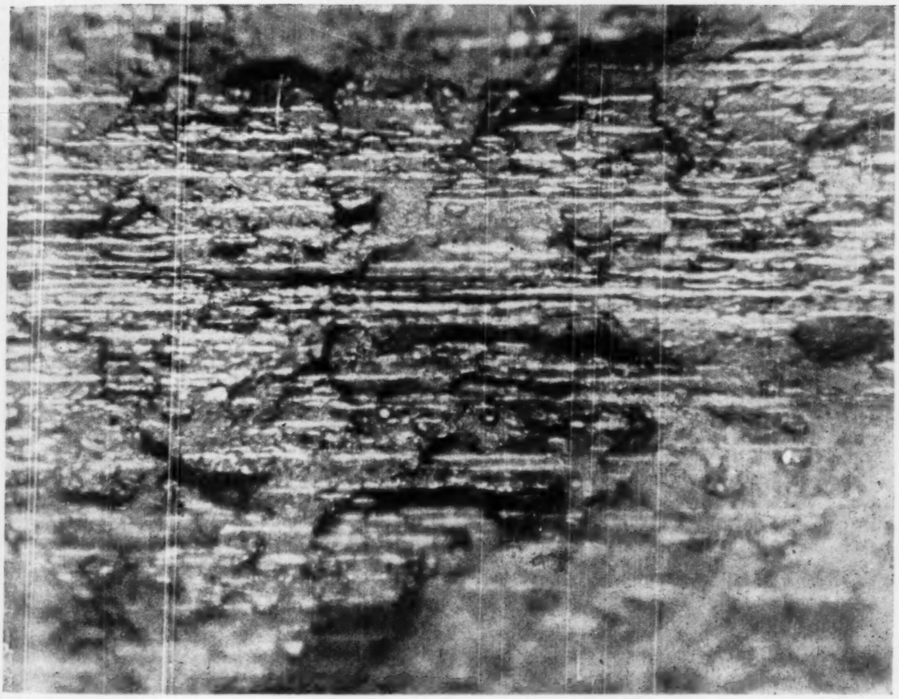
(*a*) Fracture profile (×1000).
(*b*) Fracture surface replica (×8000).

FIG. 2.—Fractographs of a Double Slant-Notch Shear Specimen of Ingot Iron Failed in Axial Tension.

closer examination of the transverse fracture at a somewhat high magnification may show some microregions which shear zones which reflect light away from the microscope eyepiece and appear less bright.



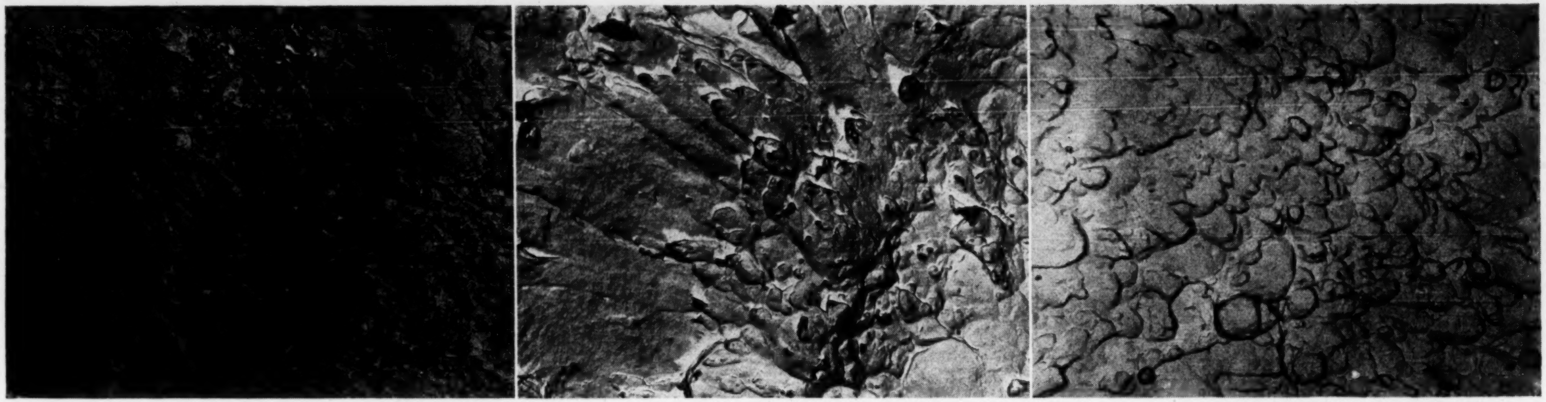
(*a*) Fracture surface (×1).



(*b*) Transverse fracture zone; darkfield illumination (×50).
FIG. 3.—Fracture Surface of a 4340 Steel Cylinder.

belong to a shear type of fracture. This is clearly seen in the fractures exhibiting parallel ridges (Fig. 3). The white streaks which appear at a moderate magnification under darkfield illumination are truly transverse fracture zones and the regions around the streaks are oblique

The electron micrographs of replicas taken from a transverse fracture surface of 4340 steel also show domains, but generally they are equiaxed and their interior is comparatively rough (Fig. 4 (*c*)). Some microregions of the fracture surface contain elongated domains and



(*a*) ×7000. (*b*) ×18,000. (*c*) ×18,000

FIG. 4.—Electron Micrographs Illustrating the Appearance of the Transverse Fracture Surface of 4340 Steel. Two-stage chromium shadowed carbon replicas.

flat decohesion areas resulting from local shear (Fig. 4 (*a*) and (*b*)).

The fracture surface of 4340 steel which is produced at a temperature of −320 F, considerably below the transition point of steel, also shows the equiaxed domains as well as the microregions with elongated domains. The fracture surface of Armco iron, however, produced at low temperatures (−320 F) exhibits river patterns or cleavage steps (**8**) but no elongated domains. Certain flat zones seem to contain a network pattern which may be identified with subboundaries (Fig. 5).

It has been shown by Tipper and others (**9, 11**) that in all transverse fractures where a pattern develops, the fracture is discontinuous and proceeds by the formation or initiation of numerous internal cracks and the material separating them is broken down by secondary cleavage or by shear to propagate the main crack front. The observations presented above give considerable support to this discontinuous theory of fracture propagation, not only in transverse modes of failure, but also in oblique shear.

DIRECTION OF FRACTURE PROPAGATION

The direction of fracture propagation in sheet steel generally is determined by means of chevron markings when the mode of fracture is of transverse type. The chevron pattern consists of a series of intermittent ridges running approximately parallel with the sheet face in the center and curving toward the surface, since the plastic flow increases toward the surface with a correspondingly lower velocity of propagation. The apexes of chevron markings thus point back to the direction from which the fracture comes.

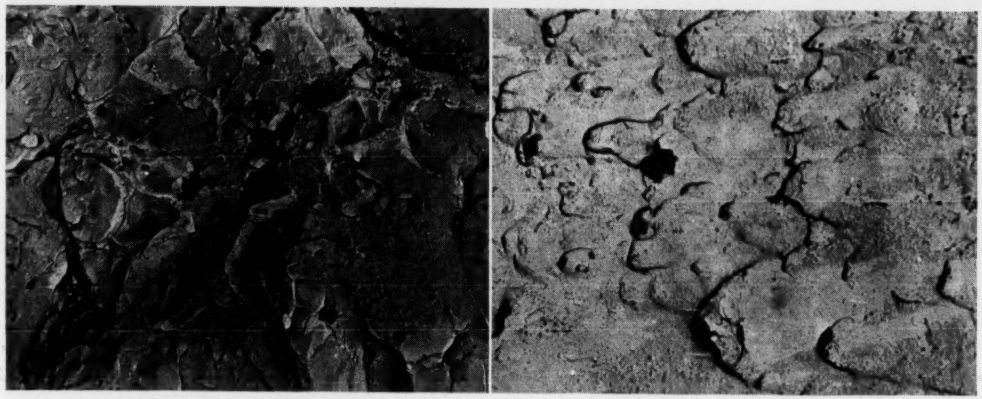
Chevron markings are not always observed on transverse fractures, particularly on thin sheets where fracture occurs rapidly at a high tangential stress and extensive shear lips are developed. Additional features are needed for determining the direction of crack propagation. Two methods are developed for this purpose: one is based on the fact that the concavity of the elongated domains seen in the electron micrographs is oriented toward the direction of the relative displacement of the other surface. The other method is based on the fact that individual or groups of secondary cracks usually run from the surface at an angle facing toward the main direction of propagation and thus a directional effect is conferred to the fracture profile.

The domain observation technique by electron microscopy is applicable to oblique shear fracture surfaces only. The elongated domains observed in certain areas of the transverse fracture surface run in all directions and cannot be used to indicate the direction of crack propagation.

A profile of the fracture face taken from a 4340 steel sheet showing chevron markings is shown in Fig. 6. The specimen was cleaned in an alkaline cleaner and electroless nickel plated to a thickness of 0.015 to 0.020 in. by suspending it in a solution of nickel chloride, sodium hypophosphate, succinic and lactic acids adjusted to a pH of 6.5 and heated to 200 F. It is then mounted in bakelite and etched with nital. It will be noted that the segments are lifted and the secondary cracks run from the surface at an angle facing toward the main direction of crack propagation as judged by means of chevron markings.

The fracture profile observation is a useful technique and can be used to determine the direction of crack propagation in many transverse fractures where chevron markings are not present.

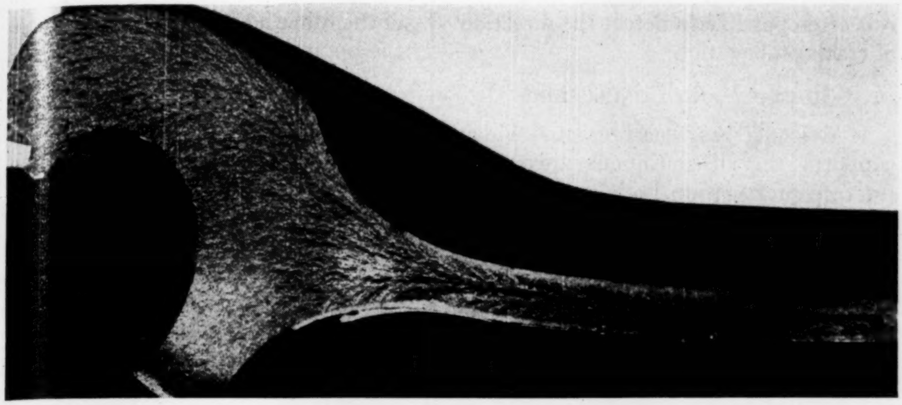
Sometimes it is difficult to find secondary cracks to evaluate the direction



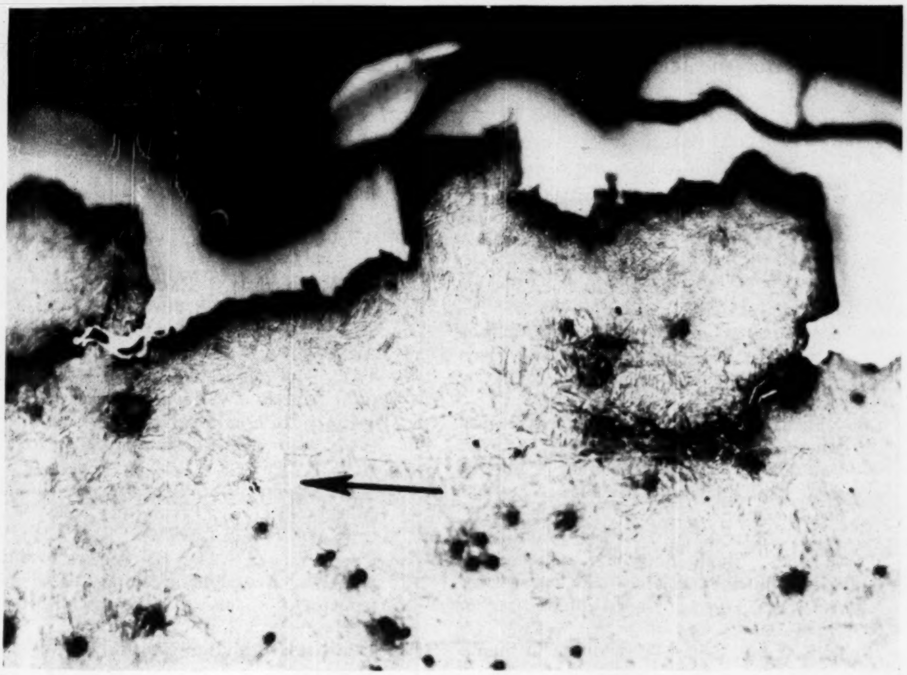
(*a*) 4340 steel (×26,000) (*b*) 4340 steel (×26,000).

(*c*) Ingot iron (×8000). (*d*) Ingot iron (×8000). (*e*) Ingot iron (×26,000).

FIG. 5.—Fractographs at Liquid Nitrogen Temperature.



(*a*) **Fracture surface (×1.5).**



(*b*) **Profile of transverse zone showing secondary cracks and direction of fracture (×250).**

FIG. 6.—Fracture Surface and Fracture Profile of 4340 Steel.

of crack propagation and occasionally a single secondary crack may be found with propagation in the opposite direction to the main crack front. However, by selecting a fracture area which contains intermittent ridges running toward the sheet surface and exploring a sufficiently large area of the fracture profile it is often possible to detect the direction of crack propagation.

SUMMARY AND CONCLUSIONS

1. Evidence has been presented which supports the discontinuous theory of fracture propagation both in transverse and shear types of failure for AISI 4340 steel.

2. Two methods of observations have been described to supplement the visual observation of the fracture surfaces for determining the direction of crack propagation:

(*a*) The examination of the elongated domains in oblique shear by electron micrography. The concavity of the domain is oriented toward the direction of the relative displacement of the other half of sheet and indicates the opposite direction of rupture.

(*b*) The examination of the fracture profiles in transverse fractures by optical microscopy at about 150 × to 250 × magnification. The secondary cracks appear to run from the surface at an angle facing toward the main direction of crack propagation and give a general directional effect to the profile.

Acknowledgments:

This paper is based on a research project conducted at the Douglas Aircraft Co., Santa Monica, on the fracturing behavior of materials. The permission of the management to publish the results is gratefully acknowledged.

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STRAIN STRENGTHENING OF TYPE 304 STAINLESS STEEL*

By Walter Hammer, Jr.,[1] Leonard Stemann,[2] and E. E. Weismantel[3]

Synopsis

A developmental program has been conducted to produce high-strength austenitic bar materials since cold-worked bars with uniform properties are not commercially available. Tensile cold-straining procedures have been used to obtain this strength improvement. Available alternative materials presented prohibitive fabrication problems that do not exist when nonheat-treatable materials are used.

Sufficient tests have been performed to indicate that this process can achieve the 100,000-psi tensile yield strength required for the current program. This strength level could be raised with additional developmental effort. Judging by the results of tests performed in this program, the compressive yield strength is estimated to be approximately 70 per cent of the cold-strained tensile yield strength. Machining, forming, and welding experiments indicated that it is no more difficult to fabricate cold-strained austenitic bar materials than other austenitic materials now in use.

In the past decade there has been a trend toward the use of high-strength and corrosion-resistant materials for designs in nuclear, rocket, and aerospace structures.

The structural details of many of these units make considerable use of cold-worked materials. These high-strength alloys are utilized as parts of welded sandwich assemblies to achieve maximum structural efficiency with minimum weight. A typical structure,[4] such as the one shown in Fig. 1, might use single corrugation sandwich panels with approximately a $\frac{3}{8}$-in. wall to wall dimension and having skin sheets of half-hard type 301 stainless steel having a minimum yield strength of 110,000 psi. The corrugated core section is of one-quarter hard type 301 stainless steel. Minimum design yield strength for the core material is approximately 75,000 psi. Because almost all such structures will operate at elevated temperatures, the effect of increasing temperatures on the properties of the materials must also be considered. Typical design curves must be developed which predict the minimum strengths of these materials over a wide range of temperature up to the point where the rupture properties become the design criteria. Such a curve for minimum yield strength is shown in Fig. 2[5-8]

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[4] Typical structure is defined as: typical of the construction and structural details necessary to meet design conditions of the specific structure used as an example herein.

[5] Data supplied by Allegheny Ludlum Steel Corp.

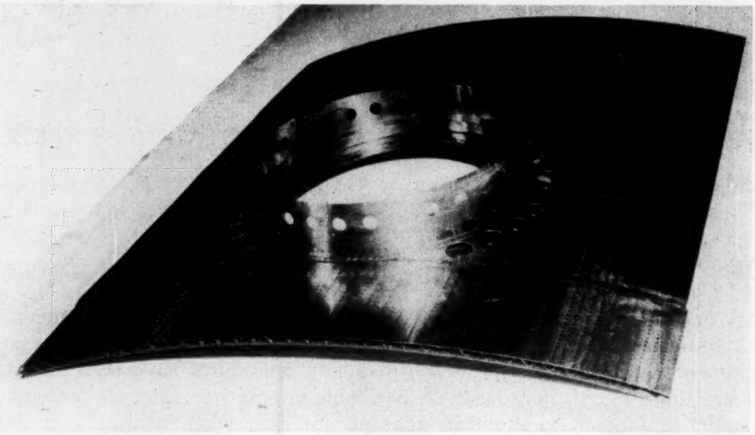

FIG. 1.—Typical Welded Sandwich Construction Using High-Strength Alloys.

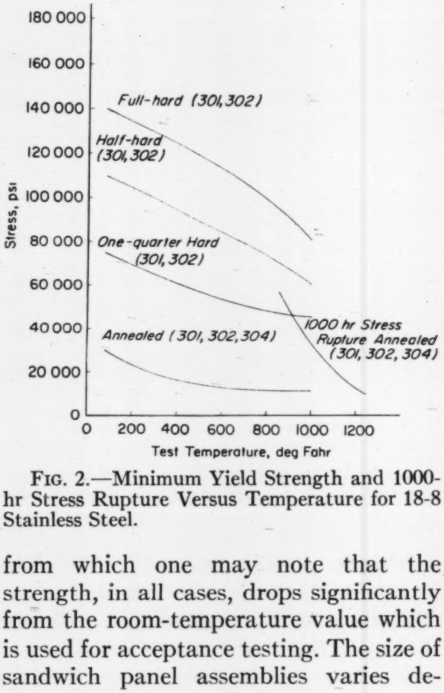

FIG. 2.—Minimum Yield Strength and 1000-hr Stress Rupture Versus Temperature for 18-8 Stainless Steel.

from which one may note that the strength, in all cases, drops significantly from the room-temperature value which is used for acceptance testing. The size of sandwich panel assemblies varies depending upon the limitation of sheet sizes available for the fabrication. Where panel sections must be joined, they are welded to machined stainless steel joining bars in the manner illustrated in Fig. 3.

These bars joining the sandwich panel sections were, at one time, designed to be fabricated from type 304 stainless steel. This was because the type 304 steel was readily compatible with type 301, and such weldments required no post-welding heat treatments. Since the joining bars were generally lightly loaded, initial design conditions would tolerate the properties of the annealed material. However, as design conditions changed, weight became more of a problem. Neither the strength of the sandwich material nor the properties of fusion weld joints limited structural design as much as did the joining bars made from commercially available material. Referring again to the series of design curves, one can see that types 301 and 304 stainless steel when annealed have estimated minimum yield points at room temperature of approximately 30,000 psi. Going on to elevated temperatures, this low value soon drops to 12,000 psi at approximately 700 F which

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[6] Air Force Technical Report No. 6517, Part 2, "Determination of Properties of Sheet Materials at Elevated Temperatures."

[7] Report on the Elevated Temperature Properties of Stainless Steel, Am. Soc. Testing Mats., *ASTM STP No. 124* (1952).

[8] The Budd Co., Design Data—Nuclear Engineering Department.

is a temperature commonly encountered in designs using cold-worked alloys. Because of this, the use of cold-drawn bar stock might be considered. Cold-drawn type 302 stainless steel is available in bars up to $\frac{3}{4}$ in. in diameter and can be obtained on special order with a minimum yield strength of 100,000 psi. Primarily because of the effect of welding heat on the material properties and because many of the joining bars used in such designs are of a size that would preclude the use of this $\frac{3}{4}$-in. maximum, the use of this commercially available material is prohibited. We first approached several of the major steel producers to determine whether cold-rolled stainless steel could be procured on special order with the required yield strength for sizes up to $1\frac{1}{2}$ by 3 in. in section. After receiving negative replies to our inquiries, we were forced to consider other alternatives.

The use of other materials such as precipitation hardening or heat-treatable stainless steels must be considered, but such usage is limited by the relative thermal expansion coefficients of the mating materials and the resultant thermal stresses induced in the structure because of the expansion differences. The use of some of these alloys also requires postfabrication heat treatments which are not practical due to the size, geometry, and required tolerances of the structure and the properties of the cold-worked sheet alloys used in that design.

On this program, the work-hardening obtained in the tensile straining of austenitic stainless steels was used to obtain the required properties on desired bar sizes. The strengthening of lengthened tension specimens to true stress levels was evaluated as a function of per cent strain, specimen geometry, specimen length, and subsequent aging.

Machineability of strained bars and possible distortion in machining was also studied. The properties of weld joints in cold-worked austenitic steels were studied subsequent to the work under discussion. The extent of the heat-affected zone and related mechanical and metallurgical characteristics are described in a paper presented to the American Welding Society.[9]

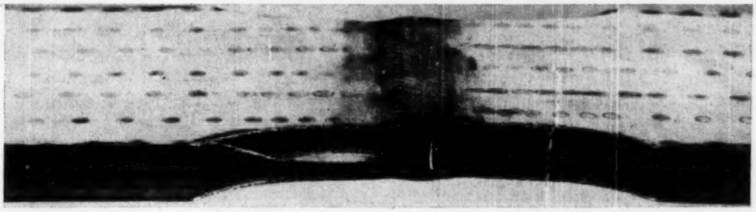

FIG. 3.—Typical Weld Joint.

TEST PROCEDURE

Phase I—Determination of Straining Characteristics of Type 304 Stainless Steel and the Effect of Specimen Geometry on the Straining Properties:

Using standard 0.505-in. diameter tension specimens, it was possible to evaluate the uniformity of straining and the hardness properties across the specimens. Figure 4 is a plot of typical hardness values obtained through the center of failed tension specimens. Two specimens are shown to illustrate that the hardening in the unnecked areas is quite similar even though the ultimate strength varied considerably. The high hardness values obtained in the necked area are not useful data for this immediate program, but the uniformity of hardness and the strengthening of the unnecked area indicated that required design properties might be obtained by the procedures that will be discussed.

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[9] L. Stemann and E. E. Weismantel, "Weld Strength and Dimensional Stability of Cold Worked Stainless Steel," *Welding Journal Research Supplement*, Oct., 1960.

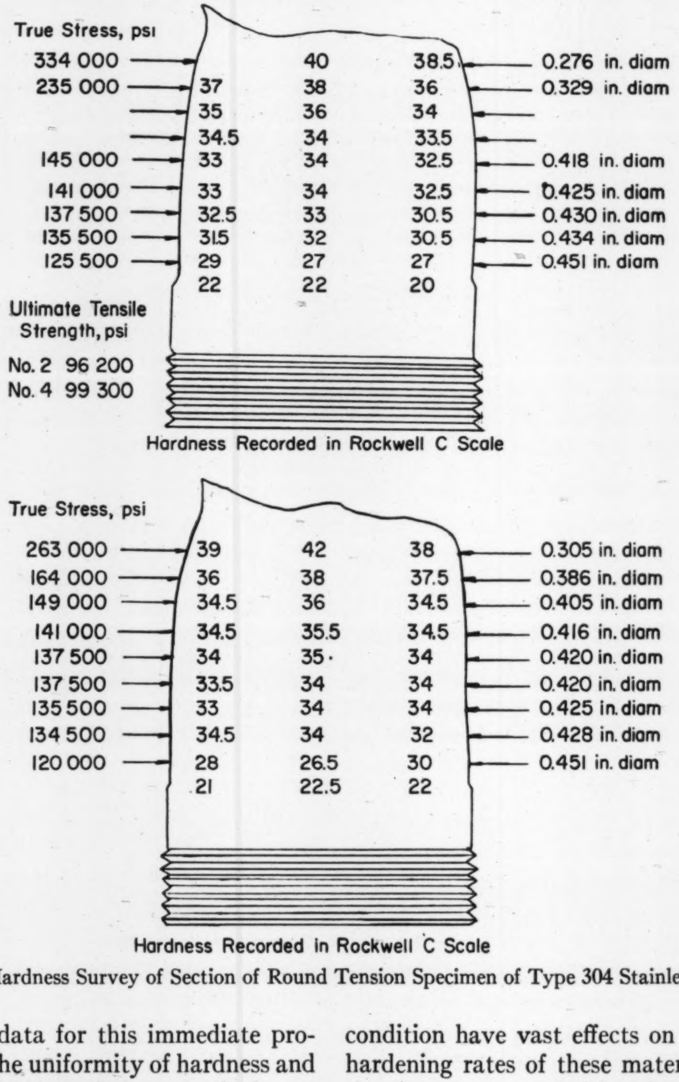

True Stress, psi
334 000
235 000
145 000
141 000
137 500
135 500
125 500
40 38.5
37 38 36
35 36 34
34.5 34 33.5
33 34 32.5
33 34 32.5
32.5 33 30.5
31.5 32 30.5
29 27 27
22 22 20
0.276 in. diam
0.329 in. diam
0.418 in. diam
0.425 in. diam
0.430 in. diam
0.434 in. diam
0.451 in. diam
Ultimate Tensile Strength, psi
No. 2 96 200
No. 4 99 300
Hardness Recorded in Rockwell C Scale
True Stress, psi
263 000
164 000
149 000
141 000
137 500
137 500
135 500
134 500
120 000
39 42 38
36 38 37.5
34.5 36 34.5
34.5 35.5 34.5
34 35 34
33.5 34 34
33 34 34
34.5 34 32
28 26.5 30
21 22.5 22
0.305 in. diam
0.386 in. diam
0.405 in. diam
0.416 in. diam
0.420 in. diam
0.420 in. diam
0.425 in. diam
0.428 in. diam
0.451 in. diam
Hardness Recorded in Rockwell C Scale

FIG. 4.—Hardness Survey of Section of Round Tension Specimen of Type 304 Stainless Steel.

In addition, initial studies on material of similar chemical analysis but having varying rolling histories resulted in data showing true stress as a function of total strain (Fig. 5). It is quite apparent that both chemical composition and material condition have vast effects on the work hardening rates of these materials. Also the true stress carried by the unnecked bar materials differs markedly from the apparent stress calculated from the original cross-section. The chemical composition and starting condition for

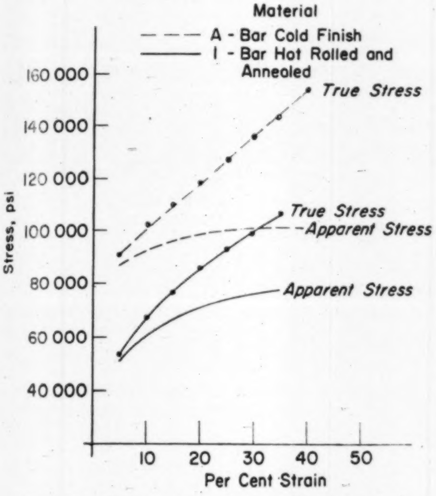


FIG. 5.—Comparison of True Stress and Apparent Stress as a Function of Per Cent Strain.

TABLE I.—ANALYSIS OF MATERIAL USED FOR INITIAL COLD-STRAINING EXPERIMENTS.

Specimen	Nominal Dimension, in.	Chemical Composition, per cent	As-Received Condition	Strain Required for Minimum 100,000 psi Yield, per cent
A, B, C, D........	1 by 1	carbon, 0.04; manganese, 0.96; phosphorus, 0.030; sulfur, 0.0040; silicon, 0.60; nickel, 9.05; chromium, 18.34	cold finished	15 to 18
Nos. 1 and 2......	⅝ by 2	carbon, 0.05; manganese, 0.63; phosphorus, 0.028; sulfur, 0.015; silicon, 0.44; nickel, 10.85; chromium, 18.72	hot rolled and annealed	28
G...............	¾ by 2	carbon, 0.05; manganese, 1.24; phosphorus, 0.025; sulfur, 0.019; silicon, 0.44; nickel, 9.40; chromium, 18.09; molybdenum, 0.15; copper, 0.15	hot rolled and annealed	35
H, I, J, K, L.....	1¼ by 2½	carbon, 0.041; manganese, 1.23; phosphorus, 0.019; sulfur, 0.011; silicon, 0.60; chromium, 18.52; nickel, 9.26	hot rolled and annealed	35

materials used in this program are shown in Table I.

Bars were strained in the as-received condition both with and without reduced cross-sections to determine the most reliable configuration for uniform strain. Bars ranging in size from 1 by 1 by 34 in. to 1¼ by 2½ by 168 in. were strained at The Budd Co. and in the testing laboratories of Swarthmore College.

After each straining program was completed, the properties of the cold-worked bars were evaluated in the longitudinal direction using standard round tension specimens cut from the test bars.

Test Results.—The evaluation of straining characteristics of stainless steel using true stress-strain curves of various heats and conditions revealed large variations in the amount of strain necessary to obtain the 100,000-psi minimum yield required for this project. This strain varied from 18 per cent to 40 per cent as shown in Fig. 6.

The effect of alloy ratios and prior processing history is illustrated by the large variations in straining characteristics. However, the lack of a sufficient number of different heats prevents an accurate compilation of data to define the principal reasons for the different straining characteristics in these materials. Hardness studies performed on

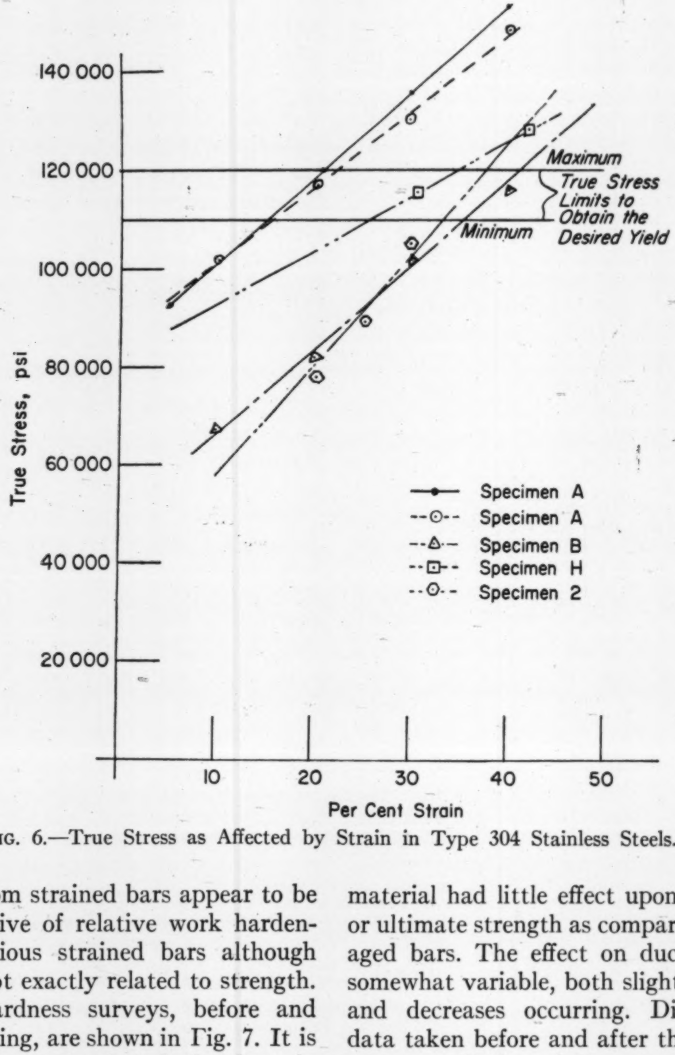


FIG. 6.—True Stress as Affected by Strain in Type 304 Stainless Steels.

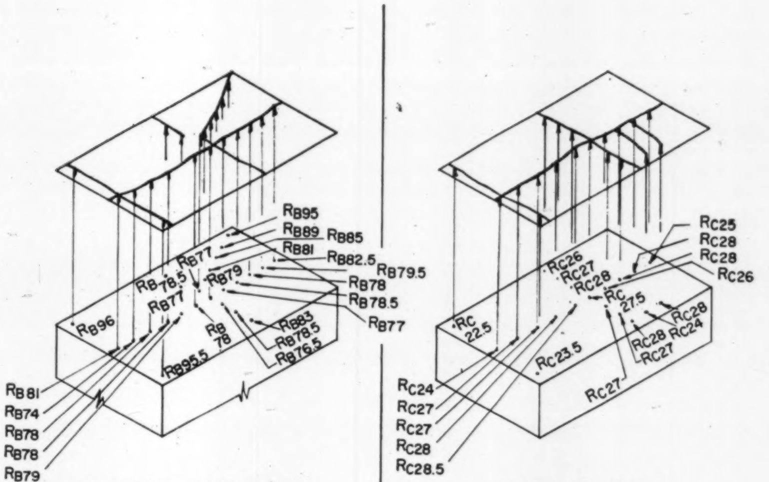
sections from strained bars appear to be representative of relative work hardening of various strained bars although they are not exactly related to strength. Typical hardness surveys, before and after straining, are shown in Fig. 7. It is worth while to note that the hardness in the center of tensile strained bars becomes slightly higher than that of the outer surfaces. This differs significantly from cold-rolled materials where the major hardening is on the outer surfaces.

Since these bars are to be subjected to service at 700 F, it had been necessary also to evaluate a stress stabilizing treatment of 2 hr at 800 F to ensure dimensional stability.

Stress stabilizing of the cold-strained material had little effect upon the yield or ultimate strength as compared to nonaged bars. The effect on ductility was somewhat variable, both slight increases and decreases occurring. Dimensional data taken before and after the thermal treatment indicated that the relaxation in length of aged specimens was as expected and ranged from 0.08 to 0.193 per cent. This was of sufficient magnitude to require a post-straining stabilizing treatment for production parts. Stress-strain curves for typical strain hardened material are shown in Fig. 8.

Phase II—Fabrication Study:

Subsequent to the development of the required strength levels from these


RB95
RB89
RB85
RB81
RB82.5
RB79.5
RB77
RB78.5
RB79
RB78
RB78.5
RB77
RB96
RB77
RB 78
RB83
RB78.5
RB76.5
RB95.5
RB81
RB74
RB78
RB78
RB79
RC25
RC28
RC28
RC26
RC26
RC27
RC28
RC 27.5
RC28
RC24
RC 22.5
RC23.5
RC28
RC27
RC27
RC24
RC27
RC27
RC28
RC28.5

Bar G Annealed (3/4 x 1 3/4)
Yield Strength - 40 500 psi
Hot Rolled and Annealed

Bar G-1 (3/4 x 1 3/4)
Yield Strength - 115 000 psi
Strained 40 per cent
Hot Rolled and Annealed

FIG. 7.—Study of Hardness of Typical Bar Cross-Sections Before and After Cold Tension Straining.

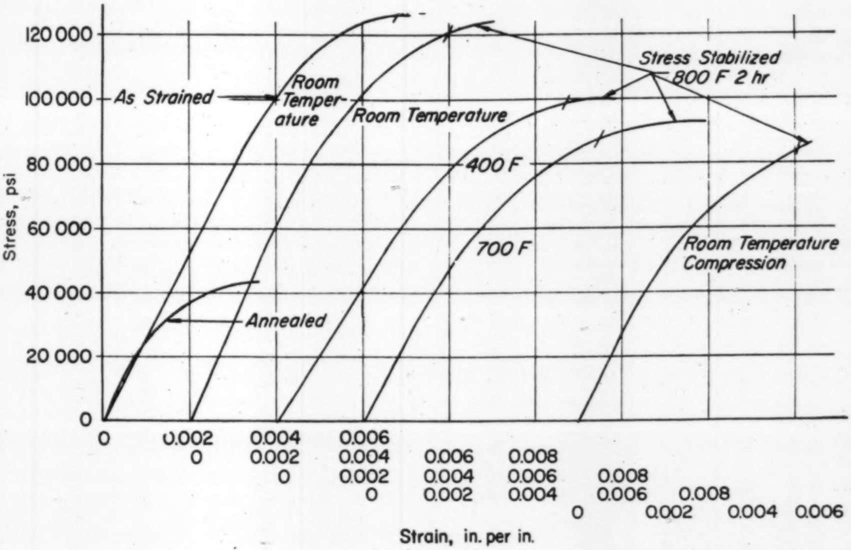


FIG. 8.—Typical Stress-Strain Curve, Longitudinal Direction, for Prestrained Type 304 Stainless Steel.

materials, a full-size quadrant representative of an actual joining bar application was machined from strain bars in a manner shown in Fig. 9. Full-size cross-sections were also machined to demonstrate machineability (Fig. 10), and were used for tension testing with welded skins in another program. No fabrication problems were detected in dimensional stability, machining, or welding, and the process was then adopted for use on a major engineering test structure.

performed using the stress calculations rather than any prescribed total strain. (This true stress is in itself a proof load value which verifies the ability of the structure to achieve desired strengths.)

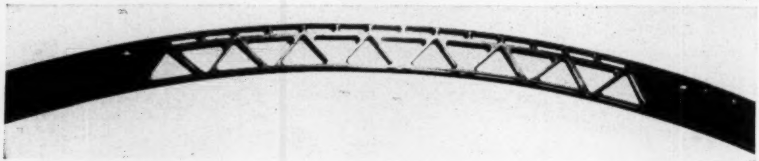


FIG. 9.—Section of Full-Size Quadrant Fabricated to Demonstrate Machinability. Both sides shown.

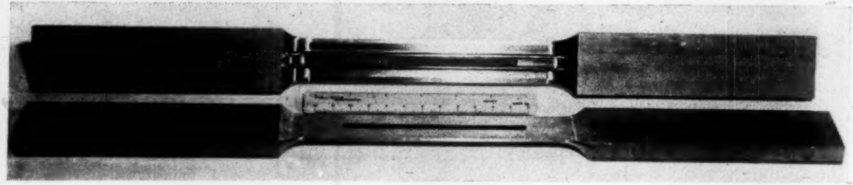


FIG. 10.—Full-Size Cross-Section Showing the Machined Geometry of Each Side.

SUMMARY AND CONCLUSIONS

The results obtained on the foregoing program indicate the practicability of tensile straining type 304 stainless steel to obtain the desired properties in the bar material. On the basis of these tests, a 100,000-psi minimum room-temperature yield strength seems readily obtainable. It is probable that, if necessary, significant additional increases in strength, over those obtained here, could be obtained with the procedures used on this program.

The use of a true stress versus strain plot will facilitate straining operations and keep variations in straining characteristics to a minimum. Good uniformity of tensile properties was obtained in long bars (14 ft) and shorter bars, without necking or nonuniform yielding regardless of per cent strain. Because of variations in work hardening rates, tensile straining operations are best

The specimen cross-sectional geometry (round or square) apparently did not have any detrimental effects on straining properties, probably because of the inherent high ductility of the material.

Aging strained bars apparently has little value in increasing stress levels above those obtained by straining. However, the measured dimensional relaxation of prestrained specimens during an 800-F stabilizing treatment has indicated that such a treatment of strained bars is necessary to avoid the buildup of residual stresses in the design structure with accompanying dimensional instability.

Beyond this dimensional instability no other problems were encountered, using this material. Machining experiments including planer, shaper, sawing, lathe, and mill operations indicated that no problems abnormal to the machining of A-286, 19-9-DL, and other stainless steel alloys should be expected. Formability of the material appears good since there is considerable ductility remaining after prestraining. Welding experiments have also been encouraging as long as the annealing effect of the weld is recognized and allowed for in design.

Based upon the results of the foregoing test program, a process specification was prepared to cover the straining of bars for the production unit. Close engineering followup on this operation revealed that the process was reproducible in the production of massive strain-hardened bars.

DISCUSSION

MR. F. R. LARSON.[1]—The authors have presented an interesting practical application of an age-old method of increasing the strength of metals through the use of cold work. This method, most suitable for materials that have high work hardening rates and large ductilities, is becoming more and more widespread.

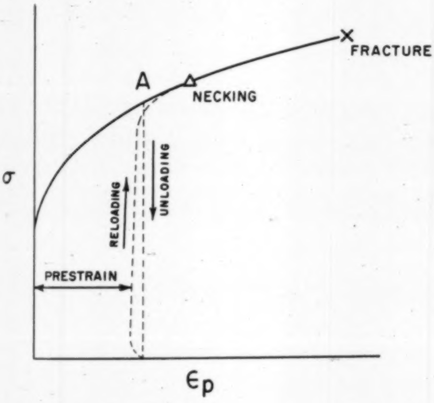


FIG. 11.—Typical True Stress, σ, - Natural Strain, ϵ, Curve.

It is apparent that the authors have recognized the advantages of considering their strength values in terms of true stress because it is computed from the instantaneous values of load and area and because it represents the new yield strength obtained for a given amount of strain hardening. However, it is unfortunate that the authors did not carry this one step further and present the data in terms of true stress - natural strain. The primary advantage of this method is that the strains are now simply additive. Furthermore, from one true stress - natural strain curve it is possible to obtain the locus of yield strength as a function of strain and also the limit of uniform extension.

Briefly, to obtain such information, continuous load and area measurements are made in a test specimen as it is pulled in tension. From the measurements, the true stress and natural strain are computed from the following well-known formulas:

$$\sigma = \frac{L_i}{A_i}$$

and

$$\epsilon_p = \ln \frac{A_o}{A_i}$$

where:

σ = true stress,
L_i = instantaneous load,
A_i = instantaneous area,
ϵ_p = natural plastic strain, and
A_o = original area.

Figure 11 is a schematic diagram of a typical true stress - natural strain curve. If a specimen is strained to fracture the true stress - natural strain curve will be a continuous function. If, on the other hand, the straining should be interrupted at a prestrain of say A, the load removed to zero, and then reloaded to continue the straining, yielding would occur when the specimen had reached approximately the true stress that was on the bar prior to unloading. So it can easily be seen from this that the true stress-strain of the authors and the true stress - natural strain curves are actually the locus of yield as a function of strain. The advantages of the true stress - natural strain method are clearly evident since it requires only one specimen to obtain the entire curve.

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[1] Chief, Mechanical Metallurgy Branch, Watertown Arsenal Laboratories, Watertown Arsenal, Watertown, Mass.

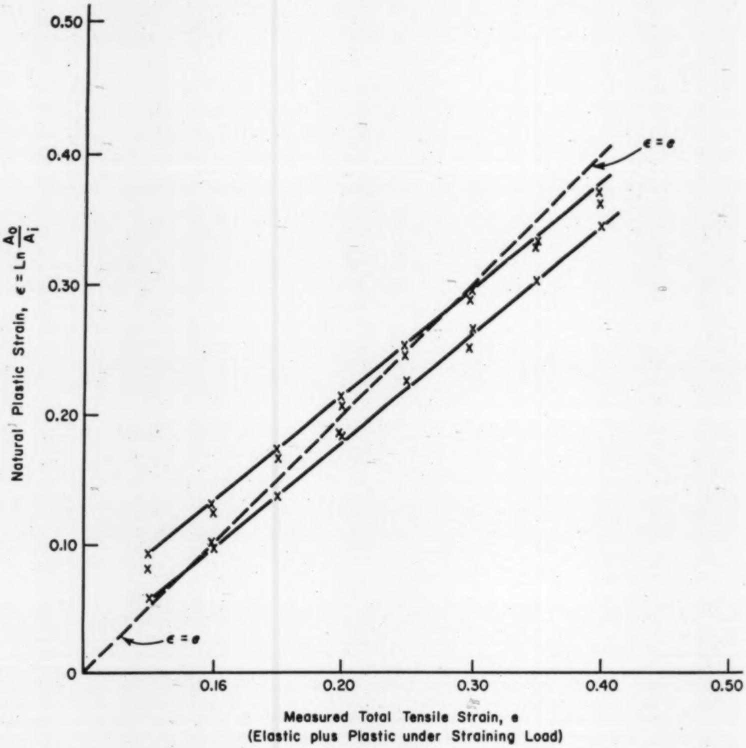

FIG. 12.—Relationship Between Observed Tensile Strain and Calculated Natural Plastic Strain for Tensile Strained Hot Rolled - Annealed Stainless Bars. Representing four bars.

Of great concern in tensile type deformation for cold working is the limit of uniform extension. This can be determined fairly accurately from the true stress - natural strain method since

$$\sigma = S_1 \epsilon_p{}^n$$

where S_1 = strength coefficient and n = strain hardening exponent = uniform strain.

A simple log-log plot of σ versus ϵ where a measurement of the slope of this plot is made will predict the approximate amount of uniform natural strain which, of course, can be converted to extension.

MESSRS. WALTER HAMMER, JR., LEON-

AR STEMANN, AND E. E. WEISMANTEL (*authors' closure by letter*).—The validity of Mr. Larson's comments regarding the relationship between apparent stress, true stress, tensile and natural strain is acknowledged. Actually in the reported development program the change in area with increasing loads was continually monitored including the calculation of strain along with the tabulation of observed strain. As we had indicated in this paper, this method is considered most positive in achieving tensile strained bar of any predetermined minimum strength. The true stress - tensile strain data have been plotted in the illustrative diagrams to present a comparison with commonly recognized apparent strain data.

In the light of Mr. Larson's comments, we have presented an additional plot showing the values of calculated natural strain as they are related to the observed (apparent) tensile strain (Fig. 12). For the degree of strain studied in this program, the calculated values approximate the observed strains with reasonable tolerance. It is because of this close approximation that we prefer to offer our data in terms of observed strain.

From this it is evident that the natural strain - measured total strain plot does not appear to intercept zero. In addition, it appears that the slope of the measured curve deviates from the plot of $\epsilon = e$. The actual reasons for this behavior cannot be reliably determined. Since all area and strain measurements were made while the system was under load, the observed strain includes an amount of elastic strain which was dependent upon the load associated with the degree of strain recorded. In addition, it must be recognized that the strengthening reported here was influenced by other factors which probably affected the variability of straining data from heat to heat of steel, as well as the intercept and slope of the natural plastic strain - total tensile strain plot. Primary strengthening was most accomplished through strain hardening common to all single-phase systems. However, since these steels are to some degree metastable austenite, it is expected that some minor transformation of austenite to martensite would be associated with the deformations used for these studies. The degree to which this occurred is not known since no such evaluation was made during the program, but such transformation would be expected to cause divergence from purely single-phase behavior.

Referring to Mr. Larson's Fig. 11, it should be pointed out that all tensile straining was carried on below the incidence of necking and sectional measurements were made throughout the length of the specimen to insure the uniformity of strain.

In accordance with Mr. Larson's suggestion, simple log-log plots can be prepared of stress versus natural strain data for the information available on these materials. The resultant slopes of these plots range from 0.25 to 0.40. This would indicate that the straining was approaching the limit allowable for uniform strain at the extremes evaluated on this program. It is not known at this time whether this was actually the case since no further testing was performed with greater strains because the objectives of the program were already exceeded. However, since more than a dozen large bars (14 ft long by $1\frac{1}{2}$ in. by 3 in. in section) were subsequently strained in this manner with no difficulty with regard to necking, one would assume that some additional plastic strain could be tolerated before nonuniform strain conditions would be encountered.

SOME EFFECTS OF SECTION SIZE AND TESTING CONDITION ON TENSILE PROPERTIES OF ZIRCALOY 2 AT 600 F*

BY S. L. AMES[1]

SYNOPSIS

A survey has been made of tensile properties at 600 F of annealed Zircaloy 2 in a number of mill product forms. Properties varied over quite a wide range. Some of the variations can be attributed to differing degrees of directionality developed in processing. In hot-rolled strip, transverse specimens had lower tensile strengths, higher yield strengths, and greater ductility than longitudinal specimens. Other products showed corresponding effects depending on process history. Vacuum annealing, as opposed to air annealing, caused a deterioration in tensile strength. Correlation of properties with oxygen impurity was generally poor although in hot-rolled strip there was a trend toward higher yield strength with increasing oxygen.

The effects of changes in tensile test procedure on measured properties have been determined. Yield and possibly tensile strengths appear to be dependent on the type of specimens used. Flat specimens generally give higher values than round specimens while machining of flat specimens to thinner gages also results in an improvement.

The main use for Zircaloy 2 is in the reaction chambers of water-cooled nuclear reactors where it operates at about 600 F. Tensile properties at this temperature are thus of importance from an engineering design standpoint. The present paper summarizes a survey of tensile properties of the alloy at 600 F in various mill product forms frequently required by nuclear core manufacturers.

Most of the tests were conducted under the test conditions outlined by the Navy as acceptance tests for Zircaloy 2 in their nuclear propulsion program.[2] Some additional tests were performed to determine the effects of changes in test procedure on measured properties.

TEST PROCEDURE

Testing stock was from fourteen 1000-lb heats and was in the form of hot-rolled strip, sheet and rod, forged sections, and cold-rolled strip. With one exception, composition was within the limits set up by the Navy for reactor grade material (Table I). The exception (heat 9YA043) was, at 366 ppm, slightly high in carbon content and contained some free carbide particles in its microstructure. As presence of this phase in small amounts has little effect on tensile properties,[3] data on this heat have been retained as valid. Oxygen is an inevitable impurity in zirconium and in commercial materials is present in amounts in excess of 1000 ppm. Its effect on nuclear and corrosion properties of Zircaloy 2 is minor, and it is one of the few impurity elements not rigidly controlled by the alloy specification. Oxygen is a potent strengthener at room temperature, but as the temperature is raised, strengthening becomes progressively less marked.[4] In the present materials oxygen levels were 1250 to 1600 ppm as detailed in Table II.

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* Presented at the Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] Senior Research Metallurgist, Research Center, Allegheny Ludlum Steel Corp., Pittsburgh, Pa.

[2] U. S. Navy Specification MIL-Z-19859A (1959).

[3] B. Lustman and F. Kerze, "Metallurgy of Zirconium," McGraw-Hill Book Co., Inc., New York, N. Y., 1955, p. 498.

TABLE I.—COMPOSITION AND TENSILE REQUIREMENTS FOR ZIRCALOY 2 FOR NAVAL REACTORS PER MIL-Z-19859A (1959).

Major Elements:

Element	Per cent by Weight
Tin	1.20 to 1.70
Iron	0.07 to 0.2
Chromium	0.05 to 0.15
Nickel	0.03 to 0.08
Zirconium	remainder

Sum of iron, nickel, and chromium contents must fall within the range 0.18 to 0.38 per cent

Maximum Impurity Content:

Element	ppm	Element	ppm
Aluminum	75	Magnesium	20
Boron	0.5	Manganese	50
Cadmium	0.5	Nitrogen	80
Carbon	270	Silicon	120
Cobalt	20	Sodium	20
Copper	50	Titanium	50
Hafnium	200	Tungsten	100
Hydrogen	25	Uranium (total)	3.5
Lead	130	Uranium-235	0.025

Tensile Properties at 600 F:

Direction	Tensile Strength, min, psi	Yield Strength, min, psi	Elongation in 2 in., min, per cent
Longitudinal	31 000	15 000	24
Transverse	29 000	18 400	35

TABLE II.—OXYGEN CONTENTS.

Heat	Oxygen Content, ppm
8YA006	1380
8YA008	1400
8YA010	1390
8YA017	1310
8YA018	1490
8YA019	1310
8YA020	1300
8YA021	1250
8YA031	1410
8YA032	1440
8YA033	1400
8YA034	1600
8YA045	1410
9YA043	1520
Zircaloy 4	1330

Heats were vacuum melted twice in a consumable electrode arc furnace, and the resultant 12-in. diameter ingots were forged from 1750 to 1850 F to a size suitable for further processing. The forging temperature was lowered to about 1700 F after initial breakdown. Some of the stock required in heavy sections was fabricated to final form entirely by hot forging. For hot rolling, billets were preheated to 1550 F except in the hand hot rolling of large sheets where rolling was from 1300 F. In producing large sheets, forged sheet bars were initially cross-rolled to increase their width. All the hot-worked products tested were production items. The cold-rolled strip was fabricated on a smaller laboratory scale, starting with two 3-ft lengths of hot-rolled 4 in. wide strip. One of these was of the nickel-free Zircaloy 2 composition (often referred to as Zircaloy 4) and was 0.135 in. thick. The second piece was standard Zircaloy 2 and of 0.150-in. thickness. The strips were cold rolled to 0.050 in. thick in stages with intermediate anneals at 1550 F in either air or vacuum. Final annealing was in vacuum at 1550 F. Tensile properties were determined at each reduction stage.

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[4] R. M. Treco, in ASM Symposium on Zirconium and Zirconium Alloys, March, 1953, p. 254.

Before testing, all material was annealed at 1550 F for 20 to 30 min. As noted above, some of the cold-rolled material was annealed in vacuum; most treatments were in air. Surfaces were conditioned after air annealing by sandblasting and pickling in a hydrofluoric-nitric acid solution.

Tension testing procedures were as recommended in ASTM Recommended Practice E 21,[5] most of the tests being also in accordance with the more rigid requirements of the Navy specification for reactor grade Zircaloy 2.[2] The latter specification stipulates a flat-type specimen with a reduced gage thickness of 0.050 in., as shown in Fig. 1. Preparation of this specimen, even from strip and sheet products, involves an excessive amount of machine shop work, and tests using two more conveniently machined specimens were run for comparison. Dimensions of one of these, a flat-type specimen with no gage thickness reduction, are included in Fig. 1. The other was a standard round test piece of 1-in. gage length and 0.252-in. diameter. For testing strip specimens in a transverse direction and for all tests on thin gage material, tabs were welded to the ends of test blanks to give correct specimen length and/or correct thickness at the grips. Strain rate, as measured by ram pacing, was 0.05 per min throughout the test except in a few special tests. The tests were conducted on a Baldwin Universal testing machine with a 120,000-lb load capacity.

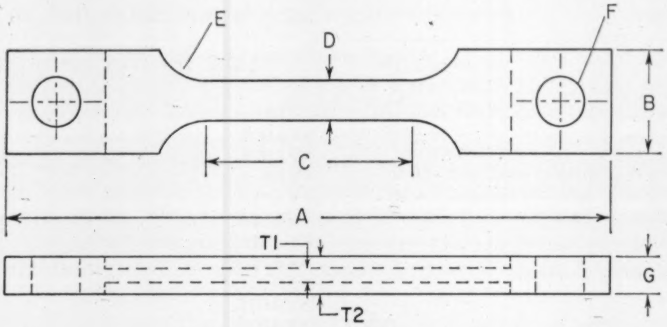

FIG. 1.—Tension Test Specimens.

	Specimen (a)[a]	Specimen (b)
A, Total length	6 in.	8 in.
B, Width at grips	1½ in.	1 in.
C, Gage length	2 in.	2 in.
D, Gage width	1 in.	½ in.
E, Radius at fillet, min	¼ in.	½ in.
F, Diameter of holes	½ in.	⅜ in.
G, Thickness at grips	0.150	as-rolled
T, Thickness in gage length	0.050 in. (T_1)	as-rolled (T_2)

[a] As required in Navy Specification MIL-Z-19859A (Ships).

TEST RESULTS

Tensile properties of a range of products are listed in Tables III to V. Most of these tests were in duplicate and mean values have been quoted. Properties varied over quite a wide range. In the longitudinal direction, for example, tensile strengths were from 29,960 to 43,270 psi, yield strengths between 17,210 and 27,080 psi, and elongation values from 25 to 43 per cent. Most of the products met the minimum requirements for Naval reactor use (Table I), the only important exception being cold-rolled and vacuum-annealed strip which had marginal tensile strength. In the strip products, directionality was well marked, transverse specimens having lower tensile strengths, higher yield strengths, and greater ductility than longitudinal specimens (Tables III, V, VI). Cold rolling as opposed to hot rolling of the strip did not greatly intensify the differences in transverse and longitudinal properties. The hot-rolled sheet had less directionality than the strip (Table III), probably due to the cross-rolling operations applied during its manufacture. Heavy forgings tended to have higher longitudinal yield strength values than the rolled products (Table IV), indicating the smaller degree of directionality developed by forging as compared with rolling.

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[5] Recommended Practice for Short-Time Elevated Temperature Tension Tests of Materials (E 21 - 58 T), 1958 Book of ASTM Standards, Part 3.

TABLE III.—TENSILE PROPERTIES AT 600 F OF HOT-ROLLED ZIRCALOY 2 STRIP AND SHEET (ANNEALED AT 1550 F).

Heat	Longitudinal Properties				Transverse Properties			
	Yield Strength (0.2 per cent Offset), psi	Tensile Strength, psi	Elongation in 2 in., per cent	Reduction of Area, per cent	Yield Strength (0.2 per cent Offset), psi	Tensile Strength, psi	Elongation in 2 in., per cent	Reduction of Area, per cent
HOT-ROLLED STRIP (8 IN. WIDE BY 0.187 IN. THICK)								
8YA006	18 940	35 300	31	58	...	...	...	...
8YA008	19 060	35 000	33	58	...	...	...	...
8YA010	19 410	36 720	35	59	...	...	...	...
8YA017	18 220	36 080	32	59	...	...	...	...
8YA018	19 500	36 270	28	59	...	...	...	...
8YA019	19 270	36 120	30	58	...	...	...	...
8YA021	17 210	35 080	32	60	...	...	...	...
8YA031	20 130	43 270	34	49	28 240	38 330	39	67
8YA032	19 890	40 010	29	44	30 450	39 340	38	64
HOT-ROLLED SHEET (96 TO 100 IN. LONG BY 40 IN. WIDE BY 0.187 IN. THICK)								
9YA043A	21 490	32 630	40	66	23 330	32 900	39	68
9YA043B	21 640	33 040	41	67	24 050	32 550	41	68

In the cold-rolling experiments use of a vacuum anneal caused a falling off in properties compared with air annealing. Longitudinal tensile and yield strengths were affected to the greatest extent. On vacuum annealing 0.090-in. gage Zircaloy 4, for example, there was a 3000- to 5000-psi drop in longitudinal yield and tensile strengths compared with the same material air annealed. There was no corresponding change in room-temperature hardness. Hydrogen contents were checked to establish the amount removed during the vacuum anneal. The difference between air-annealed and vacuum-annealed material was only 6 ppm, hardly enough to explain the observed changes in properties. A difference in duration of the vacuum

TABLE IV.—TENSILE PROPERTIES AT 600 F IN LONGITUDINAL DIRECTION OF HOT-ROLLED BARS AND OF FORGED ZIRCALOY 2 (ANNEALED AT 1550 F).

Heat	Cross-Sectional Size, in.	Flat Tension Specimen[a]				Round Tension Specimen[a]			
		Yield Strength, 0.2 per cent Offset, psi	Tensile Strength, psi	Elongation, in 2 in., per cent	Reduction of Area, per cent	Yield Strength, 0.2 per cent Offset, psi	Tensile Strength, psi	Elongation in 1 in., per cent	Reduction of Area, per cent
Hot-Rolled Products:									
8YA034........	2¼ diam	20 290	37 410	32	58	17 770	36 680	30	56
8YA045........	3.35 diam	21 690	34 890	30	54	...	...	...	...
8YA045........	2½ diam	20 560	31 200	33	63	...	...	...	...
Forged Products:									
8YA033........	7 by 5¾	22 800	32 780	32	49	23 140	32 090	35	62
8YA034........	7 by 5¾	25 640	35 810	34	52	22 085	33 200	33	60
8YA045........	4⅝ square	27 080	36 055	28	62	...	...	...	...
9YA043........	20 by 2	21 710	33 690	31	50	20 940	32 360	35	59
9YA043[b].......	30 by 2	25 730	35 220	32	44	25 050	33 170	36	62

[a] Flat specimen was as shown in Fig. 1(*a*); round specimen was of 1-in. gage length and 0.252 in. diameter.

[b] These specimens were tested in transverse direction.

TABLE V.—TENSILE PROPERTIES AT 600 F OF COLD-ROLLED STRIP.

Thickness, in.	Final Anneal at 1550 F		Longitudinal				Transverse			
	Atmosphere	Time, min	Yield Strength, 0.2 per cent Offset, psi	Tensile Strength, psi	Elongation in 2 in., per cent	Reduction of Area, per cent	Yield Strength, 0.2 per cent Offset, psi	Tensile Strength, psi	Elongation in 2 in., per cent	Reduction of Area, per cent
ZIRCALOY 2										
0.135.........	air	20	21 400	39 840	25	43	26 280	35 900	38	65
0.090.........	air	20	22 930	43 060	28	55	24 750	36 350	39	66
0.090.........	vacuum	30	19 940	39 100	29	50	24 270	33 420	40	70
0.05[a]..........	vacuum	30	22 500	35 130	32	55	24 350	32 200	38	63
0.05[b]..........	vacuum	30	21 870	33 960	36	60	24 590	30 810	41	68
NICKEL-FREE ZIRCALOY 2 (ZIRCALOY 4)										
0.160.........	air	20	18 770	35 740	33	53	25 530	32 820	40	64
0.124.........	air	20	21 670	37 380	32	55	23 340	32 660	40	64
0.090.........	air	20	23 100	37 600	34	58	22 920	32 670	39	65
0.090.........	vacuum	30	19 140	32 410	38	65	21 620	29 960	42	65
0.050[a]........	vacuum	30	21 990	32 060	38	64	23 470	29 640	42	69
0.050[b]........	vacuum	30	21 080	29 960	43	71	22 400	28 100	46	72

[a] Annealed in air at 0.090 in. thickness.

[b] Annealed in vacuum at 0.090 in. thickness.

anneal compared with air anneal of cold-rolled strip may have been partially responsible for the falling off in properties. Although nominal annealing times were not greatly different, the actual time the charge was held in a temperature range where softening could take place was longer in the case of vacuum annealing because of the necessity of charging into cold furnaces and for furnace cooling before removal. This explanation does not, however, account for the greater effect of the vacuum anneal on longitudinal than on transverse properties.

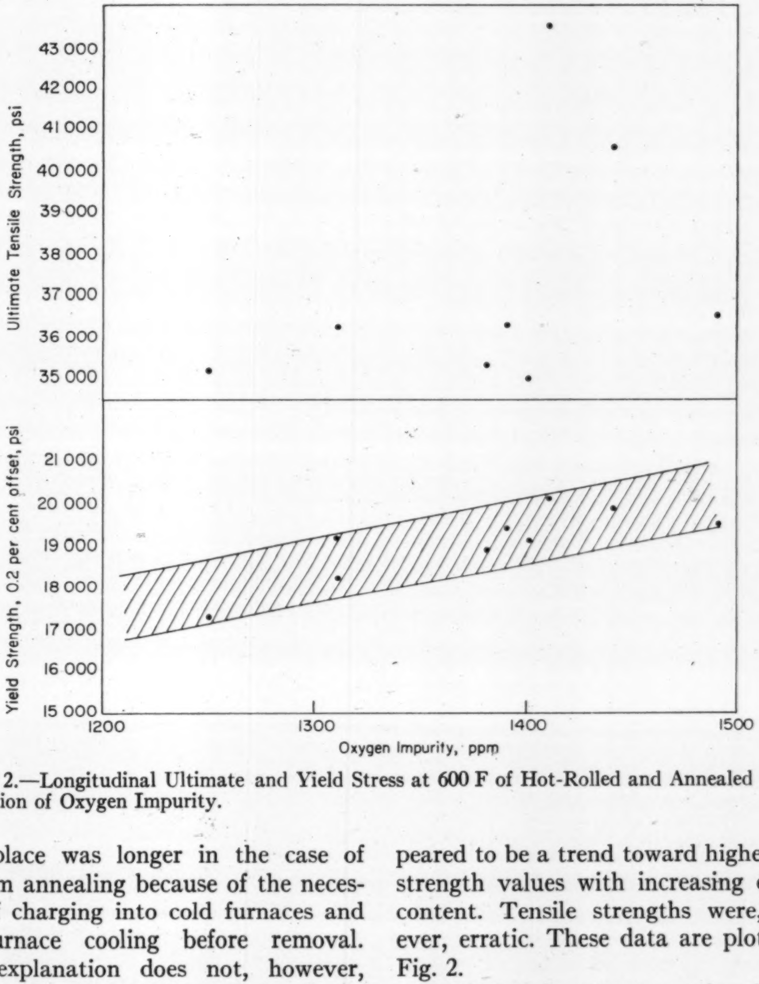

FIG. 2.—Longitudinal Ultimate and Yield Stress at 600 F of Hot-Rolled and Annealed Strip as a Function of Oxygen Impurity.

Aside from the property changes attributable to differences in directionality or to annealing conditions there was still a considerable spread in properties even in material of similar process history. Correlation of the differences with oxygen impurity content was poor. In the heats of hot-rolled strip, which had nominally identical history, there appeared to be a trend toward higher yield strength values with increasing oxygen content. Tensile strengths were, however, erratic. These data are plotted in Fig. 2.

Typical microstructures in the annealed condition of a heavy forging and of thin cold-rolled strip are shown in Figs. 3 and 4. These two micrographs represent the two extremes of structures

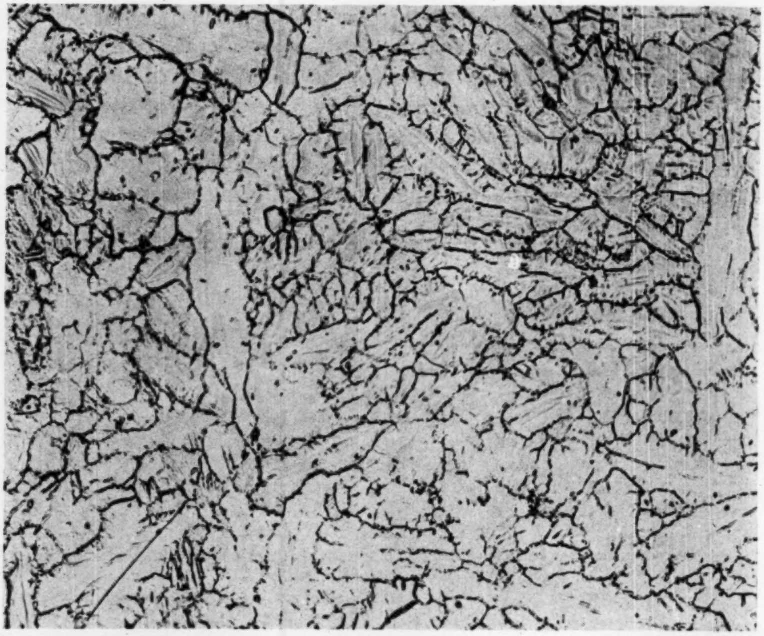

FIG. 3.—Microstructure of Heavy Sectioned Forging, 7 by 5¾-in. Section, Annealed at 1550 F and Etched in Hydrofluoric-Nitric Acid Solution (×150).

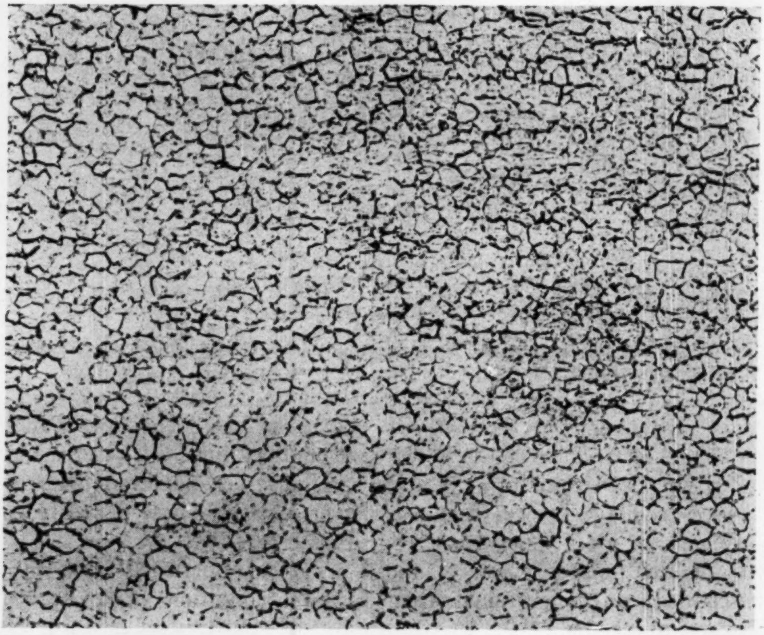

FIG. 4.—Microstructure of Cold-Rolled and Vacuum-Annealed Strip 0.05 in. Thick Annealed at 1550 F and Etched in Hydrofluoric-Nitric Acid Solution (×150).

observed. Hot-rolled sheet and bar products had structures intermediate between these, depending on the amount of work imposed in processing. ASTM grain size was about 4 in the heavy forgings but with localized regions of much smaller grain size. Further hot or cold working resulted in progressive grain refinement so that hot-rolled and annealed strip had ASTM grain size 6 to 7 while cold rolled and annealed strip had ASTM grain size 7 to 8. Cold-rolled and vacuum-annealed strip had very slightly larger grain size than equivalent material annealed in air.

TABLE VI.—COMPARISON OF 600-F TENSILE PROPERTIES USING TWO TYPES OF FLAT TEST SPECIMENS (ANNEALED AT 1550 F).

Specimen Type 1 (*a*)[a]				Specimen Type 1 (*b*)[a]			
Yield Strength, psi	Tensile Strength, psi	Elongation in 2 in., per cent	Reduction of Area, per cent	Yield Strength, psi	Tensile Strength, psi	Elongation in 2 in., per cent	Reduction of Area, per cent
22 790	34 320	40	62	18 590	31 430	36	68
22 550	34 040	43	62	19 050	32 330	38	68

[a] Specimen 1(*a*), gage length 2 in., gage width 1 in., gage thickness 0.05 in.; specimen 1(*b*), gage length 2 in., gage width 0.5 in., gage thickness as rolled (0.187 in.). Specimens were from adjacent positions in 0.187 in. thick strip.

TABLE VII.—EFFECT OF TEST SPECIMEN FORM AND STRAIN RATE ON TENSILE PROPERTIES OF ZIRCALOY 2 STRIP.

Material: Hot-Rolled and Annealed 0.187-in. Thick Strip (Heat 8YA032).
All Specimens Longitudinal.

Test Temperature, deg Fahr	Tension Specimen		Strain Rate 0.05 per min Throughout Test				Strain Rate 0.05 per min to Yield Strength, then 0.10 per min				Strain Rate 0.05 per min to Yield Strength, then 0.25 per min			
	Gage Width, in.	Gage Thickness, in.	Yield Strength, 0.2 per cent Offset, psi	Tensile Strength, psi	Elongation in 2 in., per cent	Reduction of Area, per cent	Yield Strength, 0.2 per cent Offset, psi	Tensile Strength, psi	Elongation in 2 in., per cent	Reduction of Area, per cent	Yield Strength 0.2 per cent Offset, psi	Tensile Strength, psi	Elongation in 2 in., per cent	Reduction of Area, per cent
600	0.5	0.187	17 860	35 520	31	57	17 000	36 180	31	56	17 430	38 240	31	64
		0.175	18 610	37 000	29	58	17 840	36 550	32	66	17 940	38 210	28	63
		0.100	18 070	34 870	30	62	17 710	35 330	27	56	17 030	38 090	28	56
		0.050	19 030	35 730	34	53	18 210	36 340	31	51	19 910	37 830	28	54
600	1.0	0.187	15 540	[a]	...	...	13 850	[a]	...	...	16 360	[a]	...	...
		0.175	17 940	[a]	...	...	15 950	[a]	...	...	14 410	[a]	...	...
		0.100	19 000	35 550	34	47	15 370	36 090	34	53	19 230	38 720	34	55
		0.050	17 750	34 340	32	52	17 830	35 150	34	55	17 930	36 630	31	50
Room temperature	0.5	0.187	49 210	77 430	23	37	49 370	77 200	22	35	49 590	78 140	21	36
		0.175	49 530	77 250	23	36	50 200	77 120	21	35	51 060	78 570	20	42
		0.100	49 950	77 420	29	35	50 700	77 560	21	36	50 450	77 500	19	38
		0.050	53 170	78 530	11	32	52 330	79 980	19	41	51 399	78 970	17	31

[a] Failed in grips.

Tensile and yield strength values determined from round tension specimens were usually slightly lower than those obtained from the flat specimens specified by the Navy. Similarly, in comparative tests using both the flat specimens described in Fig. 1 the Navy specimen gave higher tensile and yield strength (Table VI). Further data on this effect were obtained in tests on longitudinal specimens from hot-rolled 0.187 in. thick strip using specimens based on those shown in Fig. 1. The gage of both types of test specimens was varied, by machining, to thicknesses between 0.05 in. and the as-rolled thickness of 0.187 in. Data were also obtained on the results of increasing the strain rate after the yield stress, as is a frequent practice in many laboratories. Room-temperature in addition to 600-F properties were determined for comparison in one group of tests. Results, detailed in Table VII, showed a considerable scatter, but again suggested that yield strength values were related to gage thickness. In each group of tests where gage thickness alone was varied, the 0.05 in. thick specimens had higher yield strengths than the 0.187-in. gage thick specimens. The difference was 1000 to 2000 psi at 600 F and 2000 to 4000 psi at room temperature. The effect appeared to be due partly to the removal of the sandblasted and pickled surface of the starting strip, there generally being a significant increase in yield strength on machining only 0.006 in. from each side of the original strip. There was nevertheless a further improvement on machining to 0.05-in. thickness which could not be attributed to surface condition. Tensile strengths were less affected by variations in gage thickness in this series than in the previous tests. An apparent relationship between gage width and tensile properties was not confirmed in check tests. As expected, the increase in strain rate after the yield stress was reached caused an increase in measured tensile strength. Ultimate stress at 600 F increased by 2000 to 3000 psi on increasing the strain from 0.05 to 0.25 per min. Ductility did not appear to be affected systematically by any of these variations in test procedure.

Many investigators have examined the effects of test specimen configuration on tensile properties of the more common engineering alloys. The information accumulated has been collected and summarized in a recent review.[6] The conclusion drawn was that ductility was the only property likely to be affected by changes in test piece cross-section. Thus, the indication in the present work that the strength properties of Zircaloy 2 are affected by the type of specimen used must be regarded as unusual. It is felt that more tests, preferably in such numbers as would allow some form of statistical analysis, are desirable to confirm and study this relationship. Some work of this nature has been recently published by Goodwin and Shubert.[7] They examined 600-F properties obtained with flat and round specimens. They report no effect of specimen form on the ultimate strength but mention that in the case of yield strength values "there was a significant influence of type of sample for longitudinal specimens."

CONCLUSIONS

1. Tensile properties of annealed Zircaloy 2 in different mill product forms vary over a considerable range. Some of these variations can be attributed to differing degrees of directionality developed in fabricating to the various final forms. Transverse specimens have lower tensile strengths, higher yield strengths, and greater ductility than longitudinal specimens. The magnitude of the difference depends on the amount of directional work imposed but appears

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[6] A. Rudnick and R. L. Carson, Defense Metals Information Center Memo No. 5, Jan., 1959.

[7] J. G. Goodwin and F. L. Shubert, "Zirconium Highlights," Bettis Atomic Power Laboratory, *WAPD-ZH-25*, Oct., 1960, p. 1.

to reach a limit after which increasing work has only minor effect. Cold rolling of previously hot-rolled strip, for example, does not aggravate the directionality effects in the annealed condition.

2. Vacuum annealing, as opposed to air annealing, caused a deterioration in strength properties, particularly the ultimate strength. The reason for this was not established.

3. Apart from the above effects, there was still quite a wide spread in properties even in materials of similar process history. Correlation of properties with oxygen content was poor. Yield strength values of hot-rolled strip showed a rough trend toward higher values with increasing oxygen but otherwise no systematic variation of properties with oxygen level was observed.

4. Yield and possible tensile strengths of Zircaloy 2 at 600 F appear to be dependent on the type of tension specimen used. Flat specimens give higher values than round specimens while reduction, by machining, of the thickness of flat specimens also results in an improvement. Thus specimens of the type used for the Navy acceptance test generally give better properties than do round or as-rolled thickness flat specimens. It may be noted in this connection that ASTM Recommended Practice E 21 suggests use of tension specimens of circular cross-section wherever feasible for elevated temperature tests. On the basis of the present work material meeting the Navy tensile property requirements when tested using round tension specimens would more than meet these requirements when using the flat specimen stipulated by the Navy for all product forms.

5. Almost all the properties obtained in the present survey met the minimum requirements for Naval reactor application irrespective of product form or of tension testing conditions. The only important exception was the cold-rolled and vacuum-annealed strip which had marginal tensile strength.

DISCUSSION

MR. J. H. SCHEMEL[1] (*presented in written form*).—Mr. Ames has made an interesting contribution to a rather neglected subject of vital interest to those engaged in the manufacture and use of Zircaloy wrought products. As Mr. Ames has pointed out, the Navy specification for Zircaloy wrought products makes no allowance for the class of product produced. For example, 7-in. square forged bar must meet the same requirements as cold-rolled strip. In addition, the Navy specifies the tension test specimen illustrated in Mr. Ames' paper as (*a*) of Fig. 1. This specimen is commonly called the "H-1" and must be used for all products tested.

The Carborundum Metals Co. is currently completing a study of the effect of strain rate on the tensile properties of Zircaloy 2. For this study, we used H-1 specimens and a specimen patterned after the sheet specimen of ASTM Methods E8[2] and illustrated as (*b*) of Fig. 1 of Mr. Ames' paper. The specimens of this type were 0.150 in. thick. Our results do not agree with the comparison given by Mr. Ames in his Table VI. We find that the H-1 specimen exhibits higher elongation and ultimate strength but poorer yield strength when tested using constant strain rates. By "constant strain rate" we mean a test conducted using the separation of the gage points of the specimen as the criterion rather than the separation of the crossheads of the testing machine.

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[1] Senior Research Engineer, Carborundum Metals Co., Akron, N. Y.

[2] Methods of Tension Testing of Metallic Materials (E 8 – 57 T), 1958 Book of ASTM Standards, Part 3, p. 103.

When ram pacing such as Mr. Ames employed was used, we found no difference in the yield strengths between specimens, whereas the H-1 specimen did exhibit higher ultimate strength and elongation.

So far we have been unable to postulate a model using stresses and microstructures to explain these observations. We do feel, however, that the thin, wide test section of the H-1 specimen will certainly intensify the effect of anisotropy in the metal structure, and we are seeking a model in this line of reasoning.

In another part of his paper, Mr. Ames stated that vacuum annealing seemingly causes a deterioration in the mechanical properties of Zircaloy. We feel that the effect he illustrates in Table V is better accounted for if we consider that the air-annealed 0.090-in. thick strip adsorbed some oxygen during annealing which would raise the strength levels markedly. We may find some further evidence to support this hypothesis by observing the strengths reported for the 0.050-in. strip. Even though the two strips made in this size were both vacuum annealed after rolling to 0.050 in., the strip that was air annealed at 0.090 in. continues to be stronger than the strip vacuum annealed at 0.090 in. While perhaps some structural model might be envisioned also to account for this observation, we feel that oxygen contamination in the air anneal is the most likely explanation.

We concur with Mr. Ames' observation that specimen configuration is a major consideration in measuring the tensile properties of Zircaloy and hasten to add also that strain rate plays an important role. Our experimentation, as well as Mr. Ames' work, shows that the tension test specified in MIL-Z-19859A, using the H-1 specimen and ram pacing at the relatively rapid rate of 0.050 in. per in. of gage length per min does not always reflect the true properties of the material being tested. This is most serious because (1) it gives the engineer a distorted picture of the material being used, (2) it fails to give the producer a good index of his product, and (3) it frequently will frustrate the researcher in his quest to improve the product. There exists a need for a major overhaul of the current methods of testing Zircaloy.

MR. S. L. AMES (*author's closure*).—I would like to thank Mr. Schemel for his comments and contribution. His results on the two types of tension specimen suggest, as did our work, that relatively small differences in specimen configuration can affect measured properties. We feel that more tests are required to confirm and to establish the magnitude of these effects. Undoubtedly the effect of strain rate, emphasized by Mr. Schemel, should be included in such an investigation.

In regard to Mr. Schemel's suggestion that oxygen contamination caused the difference in properties between air and vacuum annealed strip, it must be pointed out that in testing the 0.09-in. thick strip it was necessary to machine the annealed test blanks to 0.05-in. thickness to produce the Navy tension specimen. It is very unlikely that any oxygen-contaminated layers would remain after this heavy machining operation, yet there was a considerable difference in properties between air and vacuum annealed material

EFFECT OF HEAT TREATMENT AND PLASTIC DEFORMATION ON ELECTRICAL PROPERTIES OF A MODIFIED NICKEL-CHROMIUM ALLOY

By C. Dean Starr,[1] T. P. Wang,[1] and K. A. Hofacker[1]

Synopsis

Correlation of electrical resistivity and temperature coefficient of resistance for a 75 Ni-20 Cr-2.75 Al-2 Cu alloy is presented. It is shown that a unique relation exists between these electrical properties before and after mechanical deformation or heat treatment or both.

The effect of heat treatment on the resistance and temperature coefficient of resistance of Evanohm, a 75 Ni-20 Cr-2.75 Al-2 Cu alloy, has been published.[2, 3] It has been shown that these properties can be altered appreciably by a high-temperature treatment and that the resistance and its temperature coefficient are uniquely related.[2] Once the desired electrical properties are obtained by a high-temperature heat treatment, it is of interest to determine the effect of thermal treatment and mechanical work at low temperatures on the properties of heat-treated Evanohm. This paper gives the results of such an investigation.

Thermal Stability

Nickel-chromium alloys (ASTM Specifications B 344) and nickel-chromium-aluminum-copper alloys can be heat treated. This can be done simply by holding the alloy at an elevated temperature for a prescribed period of time or by heat treating at a series of times and temperatures. When the alloy is held at elevated temperatures (500 C) to obtain the desirable properties of high resistivity (800 ohms per circular mil foot) and low temperature coefficient of resistance (±5 ppm per deg Cent) it is termed heat treatment. When the alloy is subsequently held for prolonged periods of time at lower temperatures (350 C and below) to determine how much the properties change, the process is termed stability testing. Actually, of course, they are both heat treatment processes. At or near room temperature the rate of change of electrical properties by heat treatment becomes negligible.

To measure this effect and minimize errors caused by specimen preparation, wire specimens were prepared and heat treated as outlined by Starr and Wang.[2] These heat-treated specimens were then used for stability testing. A tube furnace was used for the higher temperatures of 350 and 300 C. To prevent oxidation, cracked ammonia was piped into the tube. The hot resistance of the specimen was measured periodically, after which the specimen was removed to the front of the tube extending outside the furnace and allowed to cool to room temperature in a cracked ammonia atmosphere. The cold resistance at 20 C and the temperature coefficient of resistance from 20 to 100 C were then measured. This process was cumulatively repeated until at least 1000 hr had elapsed.

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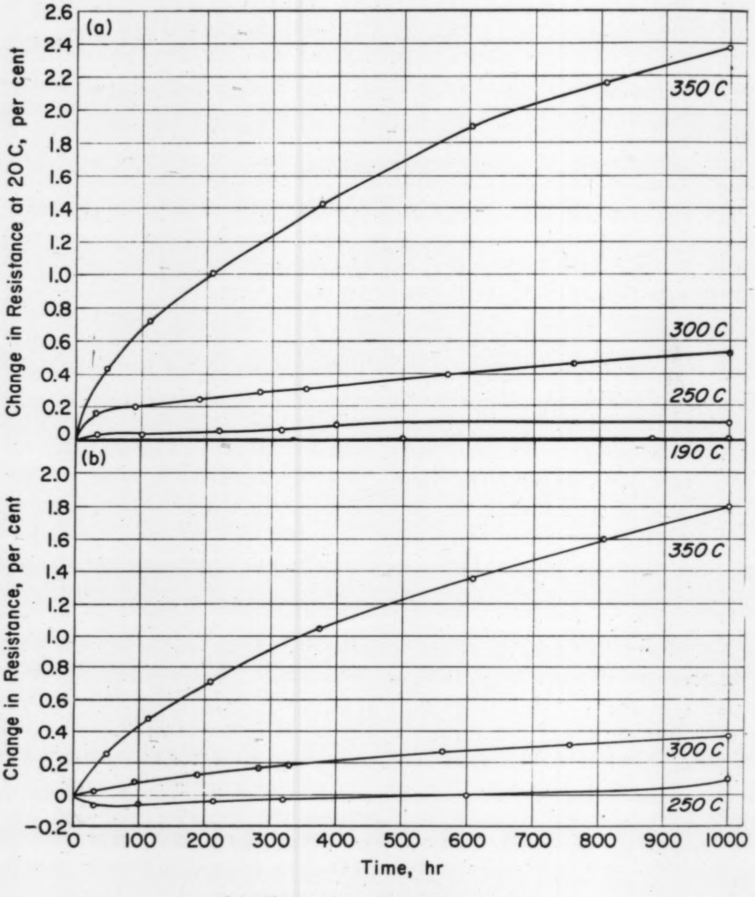
[1] Chief Research Metallurgist and Research Metallurgists, respectively, Wilbur B. Driver Co., Newark, N. J.

[2] C. Dean Starr and T. P. Wang, "The Electrical Characteristics of a Nickel-Chromium-Aluminum-Copper Resistance Wire," *Proceedings*, Institution of Electrical Engineers (London), Vol. 104, Part B, No. 17, p. 515 (1957).

[3] A. H. M. Arnold, "Nickel-Chromium-Aluminum-Copper Resistance Wire," *Proceedings*, Institution of Electrical Engineers (London), Vol. 103, Part B, No. 10, p. 439 (1956).

For the lower stability temperatures, a similar procedure was used except that the specimens were inserted in Inconel tubes immersed in a salt bath. A protective atmosphere of cracked ammonia was again used.



(*a*) Change in resistance at 20 C.
(*b*) Change in hot resistance.

FIG. 1.—Effect of Time at Various Temperatures on Change in Resistance of Evanohm.

The data for tests at 190, 250, 300, and 350 C are shown in Figs. 1(*a*) and (*b*) and 2. Figure 1(*a*) shows the change in resistance at room temperature after exposure to temperature for times up to 1000 hr. At 350 C, the resistance changes continually and shows a 2.38 per cent change after 1000 hr. At 300 C and below, the predominant change occurred during the first 30 to 50 hr, and thereafter the rate of change was uniform. For example, at 300 C the resistance increased 0.18 per cent after 50 hr, and then increased only 0.35 per cent during the next 950 hr—about 0.0004 per cent per hr. At 250 C, the maximum change was only 0.1 per cent after 1000 hr. At 190 C the change in resistance was not detectable.

Figure 1(*b*) shows similar data on the change in hot resistance. The change in hot resistance is less than the change in cold resistance at each temperature tested. Test data on hot resistance at 190 C were not obtained.

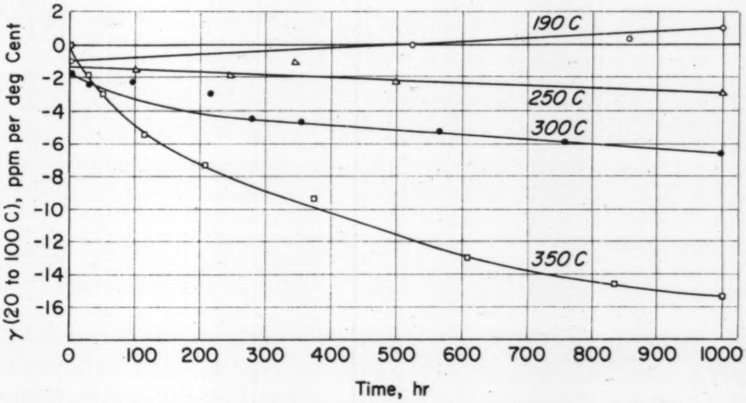


FIG. 2.—Change in Temperature Coefficient of Resistance of Evanohm After Exposure to Temperatures from 190 to 350 C for Times up to 1000 hr.

Figure 2 shows the temperature coefficient of resistance from 20 to 100 C after exposure to various temperatures. Since each specimen was heat treated separately, the initial values of the coefficient are not identical. However, all were within 2 ppm. The test results for 190 and 250 C show an increase and decrease of the temperature coefficient respectively of about 1 ppm after 1000 hr. Since this is within the accuracy of measurement, no significance can be attached to these observations. At 300 and 350 C, the temperature coefficient decreases during the test. This had been anticipated, and even the magnitude of the change can be calculated from the resistance data. Previously the resistivity and temperature coefficient of resistance were shown to be represented by the equation

$$\gamma = -0.835(\rho - 792)\ldots\ldots\ldots(1)$$

where:

γ = temperature coefficient of resistance, parts per million per deg Cent (20 to 100 C),
ρ = resistivity of the alloy in ohms per circular mil foot, and
792 = resistivity of the alloy at zero γ.

Combining Eq 1 with the definition of resistance,

$$R = \rho l/d^2\ldots\ldots\ldots\ldots\ldots(2)$$

where:

R = resistance, in ohms,
l = length of wire, in feet, and
d = diameter of wire, in mils,

one obtains

$$\gamma_1 = -0.835\left(R_1\frac{d^2}{l} - 792\right)\ldots(2(a))$$

where the subscript 1 refers to the initial state of the alloy. After stability testing at some temperature T for a time t, the relation between temperature coefficient of resistance and the resistance is

$$\gamma_2 = -0.835 \left(R_2 \frac{d^2}{l} - 792 \right) \ldots (2(b))$$

TABLE I.—COMPARISON OF CALCULATED AND MEASURED TEMPERATURE COEFFICIENT OF RESISTANCE OF EVANOHM FROM 20 TO 100 C (γ IN PPM).

Time, hr	190 C γ	γ, Calculated	Time, hr	250 C γ	γ, Calculated
0	−0.9	...	0	−2.4	...
500	0.0	−0.9	32	−1.9	−2.5
880	+0.3	−0.9	100	−1.4	−2.6
1000	+1.0	−0.9	220	−1.9	−2.7
			500	−2.4	−3.0
			1000	−2.9	−3.7

Time, hr	300 C γ	γ, Calculated	Time, hr	350 C γ	γ, Calculated
0	−1.75	...	0	0	...
29	−2.3	−2.8	50	−3.0	−2.9
93	−2.2	−3.0	115	−5.5	−4.8
190	−3.0	−3.3	209	−7.3	−6.8
282	−4.5	−3.7	374	−9.3	−9.4
352	−4.6	−3.8	611	−13.0	−12.7
567	−5.3	−4.4	808	−14.6	−14.3
758	−6.0	−5.9	1000	−15.4	−15.7
1000	−6.6	−6.4			

where the subscript 2 refers to the new state. Subtracting 2(*b*) from 2(*a*) and simplifying yields

$$\gamma_2 = \gamma_1 - \frac{0.835\rho}{100} \frac{\Delta R}{R} \ldots\ldots\ldots (3)$$

where:

γ_2 = temperature coefficient in parts per million per deg Cent between 20 and 100 C after stabilizing treatment,

γ_1 = initial temperature coefficient in parts per million per deg Cent between 20 and 100 C, and

$\frac{\Delta R}{R}$ = per cent change in resistance during stability testing.

Thus from Eq 3, any thermal treatment that causes an increase in resistance will make the temperature coefficient of resistance more negative. The magnitude of the decrease in γ can be readily calculated. For the case where the initial temperature coefficient is zero, Eq 3 reduces to

$$\gamma_2 = -6.61 \frac{\Delta R}{R} \ldots\ldots\ldots\ldots (4)$$

which reveals that a change in resistance of 0.15 per cent is necessary to change the temperature coefficient 1 ppm.

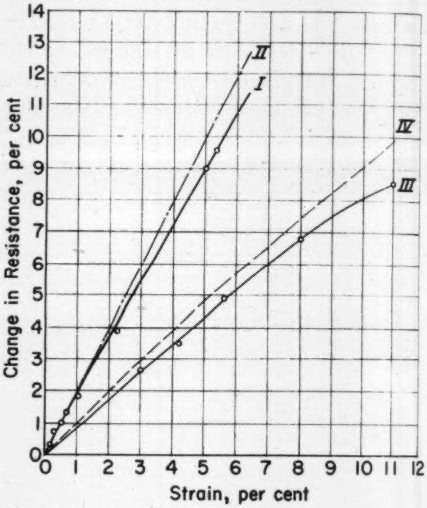


FIG. 3.—Effect of Strain on the Change in Resistance of Evanohm.

Curve I, while under load; curve II, calculated curve I; curve III, after unloading; curve IV, calculated curve III.

Table I shows the measured and calculated values of γ during stability testing at various temperatures. The agreement is within 2 ppm for all tests.

EFFECT OF UNIFORM STRAIN ON RESISTANCE AND TEMPERATURE COEFFICIENT OF RESISTANCE

All metals and alloys increase in resistance when they are uniformly deformed in tension. This is caused primarily by the increase in length and decrease in cross-sectional area of the specimen, both of which cause the resistance to increase as shown by Eq 2. When the resistivity also increases with strain, a larger increase in resistance is noted. However, nickel-chromium alloys, unlike most metals and alloys, decrease in resistivity when they are deformed. This results in a smaller increase in resistance than anticipated from dimensional changes alone. This is shown in Fig. 3 for two conditions of tensile straining. In curve I, the resistance change was measured while the specimen was under strain in an Instron tensile machine. Curve II was calculated from Eq 5 below by assuming the density and resistivity did not change during straining:

$$\frac{\Delta R}{R} = \epsilon(2 + \epsilon) \quad \ldots\ldots\ldots\ldots (5)$$

where $\frac{\Delta R}{R}$ = per cent change in resistance, and ϵ = uniform per cent strain. Since the change of resistivity with strain was not included, the calculated curve lies above the measured curve.

In the second test, wire specimens were elongated prescribed amounts in the Instron and then removed and the change in resistance per foot measured. The change in resistance is less under this condition since the length of the resistance specimen before and after straining is the same. The data from this test are shown in curve III in Fig. 3. Curve IV was again calculated by assuming no change in density or resistivity, but since the initial and final lengths of wire measured for resistance are identical, the appropriate equation is

$$\frac{\Delta R}{R} = \frac{\epsilon}{1 + \epsilon} \quad \ldots\ldots\ldots\ldots (6)$$

Again, as in the condition of the wire under load, the calculated curve is slightly above the measured curve because of neglecting the change in resistivity. In each case, however, the total change in resistance is primarily caused by dimensional changes, with the resistivity only slightly modifying the curves. Accordingly, from Eq 1, since the resistivity of the alloy is uniquely related to the temperature coefficient of resistance, uniform strain should cause only a small change in γ. To determine this, the tension specimens previously elongated a prescribed amount and removed from the Instron were submitted for temperature coefficient measurements from 20 to 100 C. These results are shown in Fig. 4 which reveals the small change in γ with strain. For example, a specimen that had an initial temperature coefficient of +17 ppm changed its coefficient only +1 ppm after a tensile strain of 1 per cent. Even after an elongation of 10 per cent the coefficient increased only 10 ppm. This behavior can be deduced from the relationship given in Eqs 1 and 2 and the assumption that the density or volume does not change during deformation. For example,

$$\gamma_i = -0.835(\rho_i - \rho_0), \quad \ldots\ldots\ldots (1(a))$$

$$\gamma_s = -0.835(\rho_s - \rho_0), \quad \ldots\ldots\ldots (1(b))$$

and

$$R_i = \frac{\rho_i l_i}{d_i^2}; \qquad R_s = \rho_s \frac{l_s}{d_s^2} \quad \ldots\ldots (2(c))$$

where the symbols refer to the same quantities given in Eqs 1 and 2 and the subscripts *i* and *s* refer to the initial and strained state.

In this test

$$l_i = l_s \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (7)$$

and

$$d_s^2 = d_i^2/(1 + \epsilon) \quad \ldots\ldots\ldots\ldots (8)$$

Substituting Eqs 2(*a*), 7, and 8 into Eqs 1(*a*) and 1(*b*) and simplifying gives

$$\gamma_s = \gamma_i - 0.835\rho_i \left[\frac{\left(1 + \frac{\Delta R}{R}\right)}{(1 + \epsilon)} - 1 \right] \ldots (9)$$

Since $(1 + \epsilon) > (1 + \frac{\Delta R}{R})$ the equation shows that the temperature coefficient will become more positive with tensile straining. Using Eq 1 to calculate ρ_i when $\gamma_i = +17$ and taking the $\Delta R/R$ and ϵ values from curve III in Fig. 3, γ_s can be calculated. These results are shown by the dotted line in Fig. 4 and show agreement within 2 ppm. This again demonstrates the unique relationship between resistivity and the temperature coefficient of resistance set forth in Eq 1.

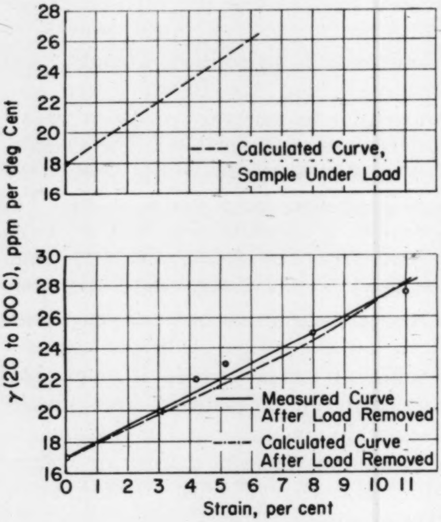


FIG. 4.—Effect of Uniform Plastic Strain on the Temperature Coefficient of Resistance of Evanohm (20 to 100 C).

The temperature coefficient of the specimen cumulatively strained and shown in curve I in Fig. 3 was not measured. However, its temperature coefficient can be readily calculated. Since in this test the resistance of the total wire was continuously measured, the appropriate equation is

$$\gamma_s = \gamma_i - 0.835\rho_i \left[\frac{\left(1 + \frac{\Delta R}{R}\right)}{(1 + \epsilon)^2} - 1 \right] . (10)$$

Using this equation and the experimental data in Fig. 3 for curve I along with $\gamma_i = +17$, the temperature coefficient as a function of strain was calculated and plotted in Fig. 4. The change in temperature coefficient with strains increases slightly faster than the unloaded specimen. In both cases, however, for strains of the order of 1 per cent the temperature coefficient increases less than 2 ppm, which is negligible for most applications.

EFFECT OF PLASTIC STRAIN DURING BENDING

When a wire of diameter *d* is wound on a mandrel of diameter *D*, the maximum strain occurs at the surface of the wire and is equal to

$$\epsilon = \pm \frac{d}{D + d} \ldots\ldots\ldots\ldots (11)$$

The + sign designates a tensile strain on the outer surface while the − sign indicates a compressive strain on the inner surface.

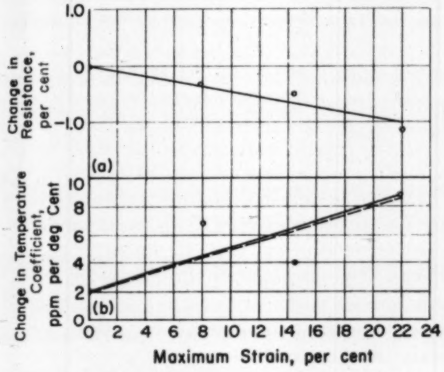
During the fabrication of a resistor, three separate effects occur which alter the resistance of an alloy. These are as follows:

1. The resistivity changes because of cold work introduced by straining. Since nickel-chromium alloys exhibit a decrease in resistivity with cold work, this effect causes a decrease in resistance.

2. During bending of a wire the inner fibers become shorter while the outer fibers become longer. Since an electrical current is conducted through all fibers, the resistivity will depend on the sum of the resistance of each fiber. This "shape factor" has been calculated for

the condition in which the density and mean length of the wire remains constant and the cross-section remains circular during bending.[4] The theory predicts that this factor causes a decrease in resistance. This has been confirmed by tests on copper and the test alloy.

3. During winding of a resistor, a tensile strain commonly called the winding tension is placed on the wire. This effect causes the resistance to increase since the dimensional changes are greater than the resistivity changes.



(*a*) Change in resistance.
(*b*) Change in temperature coefficient.

FIG. 5.—Changes in Resistance and Temperature Coefficient of Resistance of Evanohm During Bending.

Dotted line indicates calculated curve.

To minimize a combination of tensile and bending strains, the alloy was annealed at a low temperature prior to heat treating to develop a high yield strength. Wire 0.0025 in. in diameter was then wound on mandrels having diameters of 0.029, 0.015, and 0.009 in. respectively. The winding tension was kept below one seventh of the yield strength. The measured changes in resistance and temperature coefficient of resistance are given in Figs. 5(*a*) and (*b*).

The change in resistance data in Fig. 5(*a*) show that the resistance decreases linearly with the maximum strain. The maximum decrease in resistance was 1 per cent for the strains used.

The temperature coefficient of resistance increased with strain from an initial value of +2 ppm to a maximum of +9 ppm at 22 per cent strain. The intermediate values of +7 and +4 ppm are within $2\frac{1}{2}$ ppm of the curve if a linear increase in temperature coefficient of resistance with strain is assumed. If the dimensional changes caused by the "shape factor" during bending are offset by the dimensional changes caused by the winding tension, the change in the temperature coefficient of resistance can be attributed to only the change in resistivity. Thus the temperature coefficient can be calculated directly by Eq 3 where γ_2 is now the coefficient of the strained wire. Since $\Delta R/R$ is negative, the temperature coefficient increases with bending strain. As noted by the dotted curve in Fig. 5(*b*), good agreement between measured and calculated coefficients are obtained by this method.

CONCLUSIONS

1. Heat-treated Evanohm when held for 1000 hr at 350, 300, 250, and 190 C increases in resistance 2.38, 0.53, 0.10, and less than 0.01 per cent respectively. The change in temperature coefficient during this testing is small and can be accurately calculated.

2. Mechanical work such as uniform extension or bending causes an increase in resistance. Because the resistivity of nickel-chromium alloys decreases with cold work, the increase in resistance is less than that calculated from dimensional changes. The unique relationships between resistivity and temperature coefficient of resistance are shown to be applicable for electrical changes caused by straining. Up to 1 per cent strain, the temperature coefficient change is less than 2 ppm.

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[4] C. D. Brown, unpublished data, Wilbur B. Driver Co. Research Reports.

VARIATION OF POISSON'S RATIO IN THE ELASTOPLASTIC DOMAIN OF METALS

By P. S. Theocaris[1]

Synopsis

Mechanical interference of two screens of a density of 400 lines per inch is used for measurement of strain components along the two principal axes of tension specimens of several materials. With two reference screens of equal density, called "model pattern" and "master pattern," respectively, large plastic deformations can be measured accurately. Slight variation of the density of the master screen from the density of the model screen results in an initial interference pattern, called "moiré," which is added to or subtracted from the moiré pattern caused by the deformation of the specimen. This method, called the differential method, is convenient for measuring small elastic deformations as well as large plastic deformations. The accuracy of the method is dependent on the proper selection of the difference in densities.

The method is applied to the measurement of strain components of tension specimens of several aluminum alloys subjected to pure tension loading from the elastic up to the fully plastic state of deformation. The variation of Poisson's ratio of the materials tested was studied in the transition elastoplastic domain.

It was found that Poisson's ratio was constant for purely elastic loading. The change in this ratio became apparent a little before the elastic limit was reached, and its variation with strain followed a second-degree curve tending asymptotically to the limiting value of 0.5 for an incompressible material.

When investigating the variation of Poisson's ratio in the elastoplastic region of various metallic plates, dimensional changes over the entire surface of the plates must be determined. Precise simultaneous evaluation of all strain components over the entire surface of the stressed plate is difficult. Extensometric methods are confined to over-all measurements along one principal direction, while electric-resistance strain-gages, which can measure strains in several directions, are limited to the area to which the gage is attached and to small elastic strains.

The moiré method is based on the mechanical interference of two line screens, one printed on the surface of the deformed specimen and the other on a transparent reference surface superimposed on the metallic surface. Moiré fringes measure the relative displacement between the two line screens and give an over-all picture of both strain components over the entire surface of the deformed plate. Poisson's ratio can be deduced easily from these strain components at every stage of loading up to fracture of the specimen.

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Although the importance of the variation of Poisson's ratio in the transient elastoplastic region of various metals is self-evident, there appears to have been no extensive experimental investigation of this phenomenon. To the author's knowledge only Nadai **(1)**[2] discusses the continuous change in Poisson's ratio in the elastoplastic region. He introduces a variable Poisson's ratio, ν, defined as a function of the values of Poisson's ratios at the elastic and plastic regions and the corresponding constants at the elastic region and at the considered point of the stress-strain curve. He represents the variation of Poisson's ratio of a perfectly plastic material with a branch of an ordinary hyperbola, starting from the yield point and tending asymptotically to the limiting value of 0.5 for an incompressible material. He assumes that this coefficient remains constant during all elastic deformation.

The introduction of a variable Poisson's ratio in the elastoplastic region to eliminate certain difficulties encountered in problems of partial yielding is attributed to Ilyuchin **(2)**. Swainger **(3)** studied the variation in transverse contraction of mild steel under simple tensile loading and stated that transverse strain does not follow the longitudinal strain faithfully in the loading path between primary and secondary yield.

Stang, Greenspan, and Newman **(4)** measured Poisson's ratio on both flat-strip and round-bar specimens of steel and aluminum alloy. The maximum measured values of the ratio varied between 0.405 and 0.47 depending upon the material used. Gerard and Wildhorn **(5)** tested square-section specimens of aluminum alloy in both tension and compression and found maximum values well exceeding the limiting value of 0.5. The reliability of the results obtained in both cases may be doubtful. Recently Shelton and Ford **(6)** carried out tension tests in the elastic and elastoplastic regions of strain of a number of metals. Their results show true-stress versus true-strain curves and the corresponding Poisson's ratio versus true-strain relationship. The latter curves compared with theoretical curves determined by a law introduced by Nadai **(1)** show good agreement, provided the accurate values of elastic constants of the materials are known.

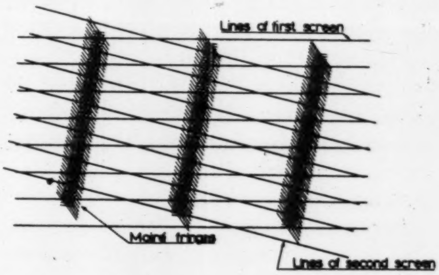

FIG. 1.—Construction of Moiré Pattern of Two Line Screens.

THEORY OF THE METHOD

This method of strain measurement requires the presence of patterns consisting of a basic unit called the "motif." The motif is repeated at regular intervals and in several directions. Two orthogonal directions are sufficient for our experiments. The superposition of two patterns of the same or different motifs results in a mutual interference known as a "moiré."

The simplest example of superposition of two line screens of equal pitch at a small angle results in a moiré with fringes running between the rows of intersections as shown in Fig. 1. It is clear from this figure that a covariant vector can be associated with each pattern, and the moiré fringes represent the covariant vector difference of the two patterns. If we apply to each screen a coordinate system of reference which has the two greatest line pitches for its covariant base vectors, then the components of each pitch are always a pair of relatively prime integers. These reference systems are called cardinal coordinate systems.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

A line screen is a one-dimensional pattern, its lattice having only one grating space of non-zero magnitude. Therefore the moiré produced by the interaction of two line screens contains only one sublattice, as shown in Fig. 1. In the case of two line screens of roughly equal intervals, in magnitude and direction, the moiré produced is given by their covariant vector difference and is, therefore, much larger than the pitches of the initial line screens. If the relative orientation of two screens is varied, the resulting moiré fringes are of maximum size when the two screens are parallel.

For measurement of strain distribution on a flat surface, an orthogonally crossed screen is printed on the surface of the metallic specimen when the material is unloaded. Although the orientation of the print with respect to the surface is not important, selection of a pattern with directions coinciding with the principal axes of the specimen may simplify the subsequent calculation. An original pattern, identical to the pattern printed on the metallic surface, is preserved by printing the same pattern on a thin sheet of film and cementing this sheet on a flat sheet of glass with an epoxy cement. The two patterns, called the "model pattern" and the "master pattern," respectively, are superimposed so that their lattices are parallel. If the pitches of both screens are the same, no moiré effect is produced through this superposition. If the pitch of one screen is changed because of the progressive deformation of the model screen, a moiré pattern is produced which measures the relative displacement between the two screens. Hence, through graphical differentiation the components of strains can be calculated.

The density of the moiré fringes greatly influences the accuracy of determination of the strain components. This density depends on the initial screen density and the magnitude of developed strains on the specimen. The density of the screen is limited by the photographic process used in printing the screens. Master screens with good contrast are obtained with a density up to 16 lines per millimeter, or a grating space of 62.5 μ. Beyond that limit the difficulties of reproduction have not yet been overcome. Therefore, this simple method is suitable for the measurement of strains in the plastic region of deformation of metals. Elastic strains produce only a few moiré fringes, which do not permit accurate determination of the elastic strain components.

For accurate measurement of elastic strains two screens with slightly different spacings are used. In this case an initial moiré is obtained which is added vectorially to the moiré produced by progressive deformation of the specimen. Selection of the proper initial moiré results in a dense moiré pattern suitable for accurate determination of the strain distribution on the specimen surface (**23**).

The method described can be applied to measure either deflections of plates (**7**) or strains in plane problems. Since this paper is concerned with the latter, a concise comparative account of the early work done on this subject follows. The first paper dealing with the intepretation of moiré fringes seems to have been published by Tollenaar (**8**) in 1945. In 1948 Weller and Shepard (**9**) used the same method to measure displacement fields. The application of the method to strain determination in plane stress problems was done by Kaczer and

Kroupa (10). In 1954 Dantu (11) introduced the intepretation of the moiré patterns in terms of the displacement components. He gave also in a series of papers a general theory of moiré for the measurement of small deformations and several applications of the method in special problems. Diruy (12) applied the same method to measurement of elastic strains by using screens of different grating space. Bromley (13) obtained similar results by using tensor notation, and Rogozinski (14) attempted to establish the theoretical foundation of the moiré method. The same method was used in papers by Crisp (15) and Vinckier and Dechaene (16) and, more recently, by Bouwkamp (17). The theory of moiré produced not only by line screens but also by other characteristic families of lines was examined by Lehman and Wiemer (18) and recently by Pirard (19). In the same paper Pirard discussed also the application of the interference screens method for the measurement of the sum of principal stresses in plane stress problems. This method was first introduced by Mesmer (20) and separately by Post (21). Another application of the method of considerable interest is its use to measure the relative dispositions and spacings of the lattice planes in the crystals of various substances, particularly metals. The method was first applied by Menter (22) and found a broad application by many specialists in this field.

Let us examine the results when the two systems have slightly different pitches. The cardinal coordinate systems related to the model pattern and master pattern are represented by κ and κ' respectively. Superposition of the two patterns before any deformation of the model gives an initial moiré resulting from the covariant vector difference of the two cardinal systems κ and κ'. If the base vectors of the cardinal system κ' are denoted by $\overset{\kappa'}{e}_{\lambda'}$, then the resulting moiré fringes are $\delta\overset{\kappa'}{e}_{\lambda'}$, given by the relation:

$$\delta\overset{\kappa'}{e}_{\lambda'} = -\overset{\kappa'}{e}_{\sigma}\, u^{\sigma}_{\lambda'} = -u_{,\lambda'} \ldots\ldots\ldots (1)$$

where the comma indicates covariant differentiation. Since system κ is cartesian, Eq 1 can be written

$$\delta\overset{\kappa'}{e}_{\lambda}{}' = -u_{\kappa',\lambda'} \ldots\ldots\ldots\ldots\ldots (2)$$

which becomes, using the usual definition of infinitesimal strain,

$$\epsilon_{\kappa'\lambda'} = \tfrac{1}{2}(u_{\kappa',\lambda'} + u_{\lambda',\kappa'}) \ldots\ldots\ldots (3)$$

$$\epsilon_{\kappa'\lambda'} = -\tfrac{1}{2}(\delta\overset{\kappa'}{e}_{\lambda}{}' + \delta\overset{\lambda'}{e}_{\kappa'}) \ldots\ldots\ldots (4)$$

Now, if the cardinal system related to the deformed grid of the specimen is denoted by k'' then, again, superposition of the deformed model pattern over the master pattern results in a new moiré given by the covariant differences $\delta\overset{\kappa''}{e}_{\lambda''}$ and, therefore, the measured strains $\epsilon_{\kappa''\lambda''}$ are expressed by

$$\epsilon_{\kappa''\lambda''} = \tfrac{1}{2}(\delta\overset{\kappa''}{e}_{\lambda''} + \delta\overset{\lambda''}{e}_{\kappa''}) \ldots\ldots\ldots\ldots (5)$$

The true strain, caused by the deformation of the specimen, is given by the covariant vector difference of the two components. Hence,

$$\epsilon_{\kappa\lambda} = -\tfrac{1}{2}(\delta\overset{\kappa''}{e}_{\lambda''} + \delta\overset{\lambda''}{e}_{\kappa''}) + \tfrac{1}{2}(\delta\overset{\kappa'}{e}_{\lambda'} + \delta\overset{\lambda'}{e}_{\kappa'})$$

$$\epsilon_{\kappa\lambda} = -\tfrac{1}{2}(\Delta\delta\overset{\kappa}{e}_{\lambda} + \Delta\delta\overset{\lambda}{e}_{\kappa}) \ldots\ldots\ldots\ldots\ldots (6)$$

where the capital delta denotes the difference of the covariant vector differences. The method thus developed is called the differential method (12). The strain components ϵ_{x_i} along the two principal directions ($i = 1.2$) of the examined pure tension specimens are expressed by the relation

$$\epsilon_{x_i} = \frac{\partial u_i}{\partial x_i} \pm \lambda_i \qquad (7)$$

where $\frac{\partial u_i}{\partial x_i}$ are the gradients of the displacement functions $u_i(x_i)$ determined by the moiré fringes in both directions, and λ_i is given by the equation

$$\lambda_i = \frac{e_i}{f_i \mp e_i} \qquad (8)$$

where e_i is the grating space of the line master screens and f_i the initial interfringes produced by each master pattern and the corresponding undeformed model pattern. The plus sign indicates a difference between the pitches of the undeformed model pattern and the master pattern of the same sign as the expected deformation of the specimen, and the minus sign indicates the reverse.

EXPERIMENTAL PROCEDURE

Three large tension specimens from each of the materials studied were constructed from plates 2 mm thick. The aluminum alloys examined were 990A, ER-5052, and 2017.

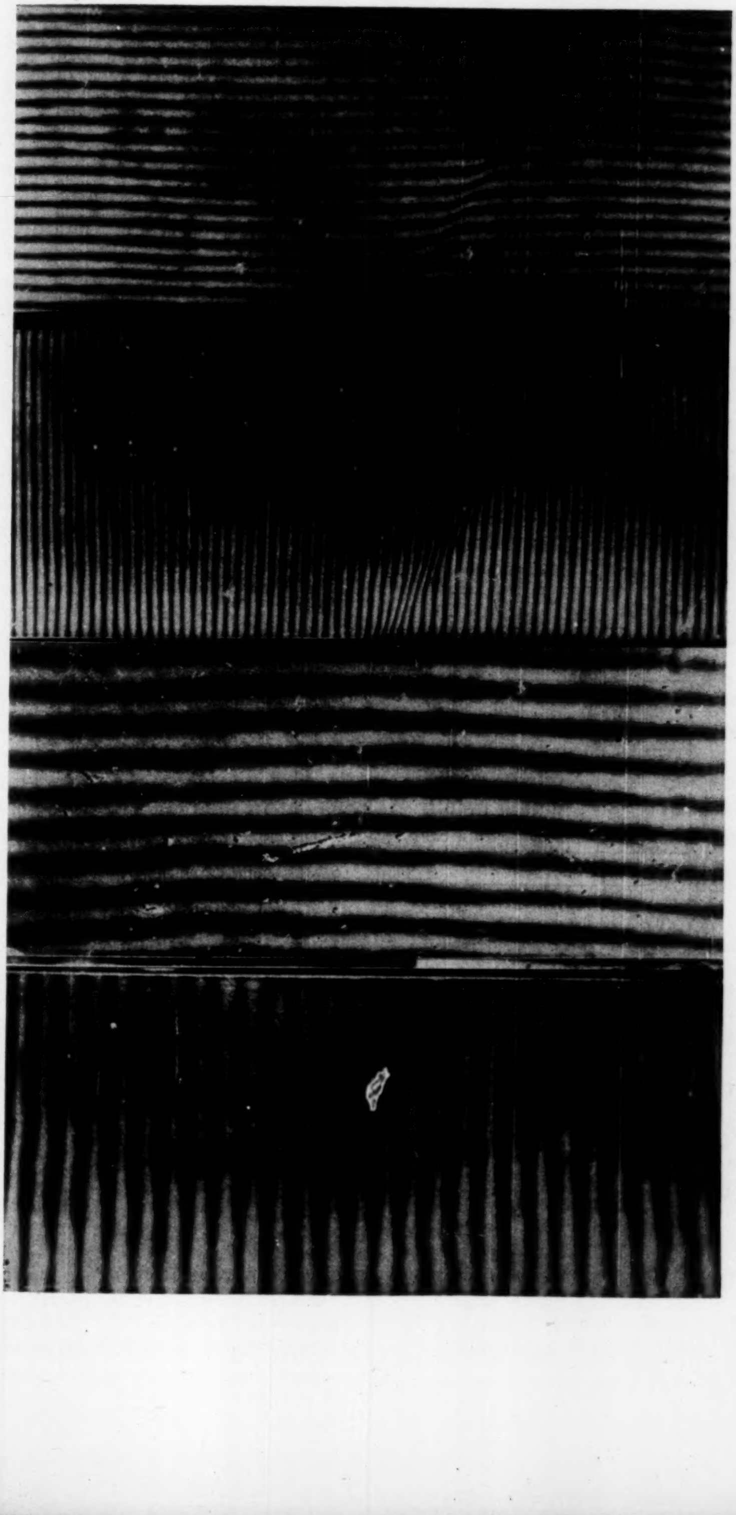
The dimensions of the specimens were such as to leave a pure tension area 120 mm square. A square screen with a grating space of 62.5 μ was printed in this area by a photoengraving process. The sides of the screen were printed parallel to the principal axes of each specimen.

Line screens of the same pitch were reproduced so that the emulsion side of these patterns coincided exactly with the portion of the pattern printed on the metallic surfaces, resulting in remarkable refinement of the moiré fringes produced.

One half of these patterns was subjected to a shrinkage treatment, the other half to a stretching treatment. The shrinkage treatment consisted of immersion in a bath of a 30 per cent solution of methanol in tap water. The time of immersion varied between 2 and 30 min and resulted in various amounts of shrinkage, down to an initial interfringe of 7 mm. The stretching treatment consisted of exposure to a closed-chamber atmosphere containing saturated vapors of the following preparation: 70 per cent of butanol with 10 per cent of methanol dissolved in 15 per cent of water by volume; 50 g of camphor per 100 ml of the above solution was also added. A layer of cotton at the bottom of the closed chamber was soaked with this preparation. The time of exposure varied from 6 to 36 hr, giving various densities of line patterns. The smallest interfringe produced by superposition of a stretched line screen with a standard one was approximately 9 mm.

The two groups of stretched and shrunken master patterns were cemented onto the surface of flat glass plates with their emulsion side away from the glass surface. The shrunken master patterns were used to measure the longitudinal extensions and the stretched master patterns to measure the transverse contraction strains at various loading steps. Selection of different master patterns for the measurement of extensions and lateral contractions resulted in a minus value in Eq 7 in both cases. Proper selection of the superimposed master pattern in the various steps of loading resulted in a value for the term λ_i which was of the same order of magnitude as the first term. The difference between these two terms gives the corresponding values of elastic strains with good precision.

The moiré patterns in both directions were photographed at each loading step with polystyrene-base film. Positives of these photographs were prepared on transparent films of the same base and on the same scale as the specimen dimensions. The time of exposure and the quantity of light were so selected as to



(a) Longitudinal elastic strain. (b) Transverse elastic strain. (c) Longitudinal plastic strain. (d) Transverse plastic strain.

FIG. 2.—Moiré Patterns on a 2017 Aluminum Alloy Specimen.

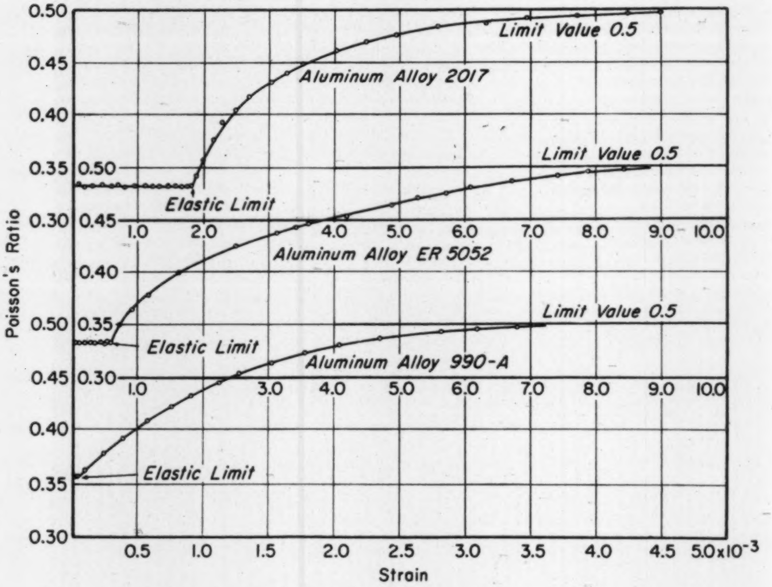


FIG. 3.—Variation of Poisson's Ratio Versus Conventional Strain.

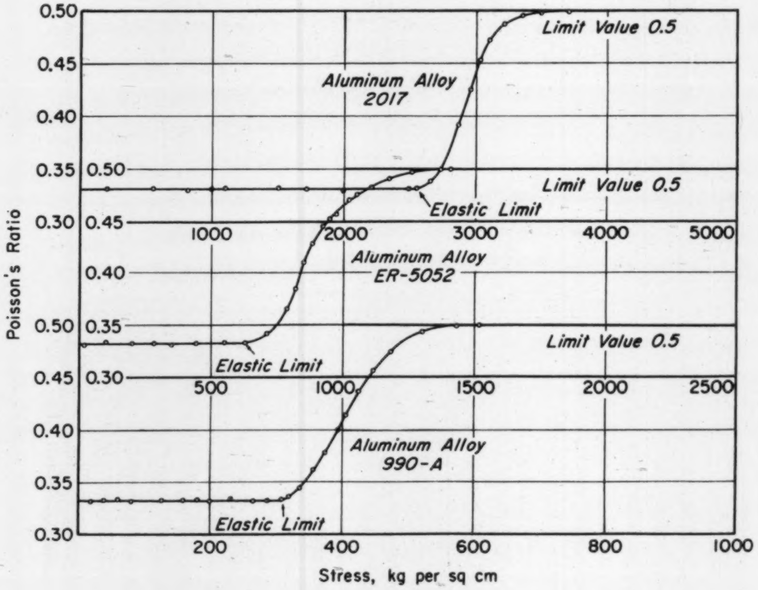


FIG. 4.—Variation of Poisson's Ratio Versus Applied Stress.

obtain a rather underexposed photograph showing only the darkest parts of the moiré fringes. Plots of the displacement curves along the two principal axes were constructed which coincided fairly well with straight lines of different slopes.

Before testing, each specimen was photographed in superposition with each master pattern used during the test in order to determine the initial moiré pattern and the corresponding interfringe f_i from which the values of λ_i were calculated. The strains were measured on a base of 100 mm in both the unloaded and the loaded specimens. The large base was used because of the uniformity of the strain field, resulting in considerable increase in accuracy of the measured strains.

Figure 2 shows moiré patterns of a 2017 aluminum alloy specimen along the two principal directions. Figures 2(*a*) and (*b*) correspond to an elastic loading step, Figs. 2(*c*) and (*d*) to a purely plastic step. Figures 2(*c*) and (*d*) also show oblique necking of the specimen.

Figures 3 and 4 show plots of variation of Poisson's ratio versus conventional strains and stresses applied to the specimens.

SUMMARY

The differential moiré method, as described in this paper, is a development of the basic method of mechanical interference of two screens applied to the measurement of strains. The method makes possible the determination of small elastic deformations as well as of plastic deformations with great accuracy, depending on the proper choice of master screens. Thus it gives a unique method for the continuous study of the state of deformation of various materials from the elastic range up to fracture.

The method is applied to the study of variation of Poisson's ratio at the transient elastoplastic domain for a series of aluminum alloys.

From this experimental study it is concluded that Poisson's ratio remains constant up to a point slightly below the elastic limit. Thereafter, its variation with strain follows a second degree curve, tending asymptotically to the limiting value of 0.5 for incompressible materials.

Acknowledgment:

The experimental study described in this paper was sponsored by the U. S. Department of the Army, European Research Office, under Contract DA-91-591-EUC-1632. The support of this agency is greatly appreciated. The author is indebted to his staff at the Laboratory for Testing Materials, Athens National Technical University, for their assistance in the project. He wishes also to express his appreciation to Dr. P. Dantu and the staff of the Laboratoire Central des Ponts et Chaussées of the Ministry of Public Works of France for their advice and help in the preparation of the master screens.

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ROOM-TEMPERATURE CREEP RATE OF MOLYBDENUM AND EFFECT OF STRAIN RATE ON TENSILE PROPERTIES OF MOLYBDENUM*

BY R. Q. BARR,[1] M. SEMCHYSHEN,[1] AND G. A. TIMMONS[1]

SYNOPSIS

The strain-rate sensitivity of molybdenum has been verified. Yield strengths were found to vary proportionately with elastic strain rate, and tensile strength was sensitive to plastic strain rates. Ductility was not markedly sensitive to changes in strain rate over the ranges evaluated. The actual strain rate occurring during early stages of load application was found to be substantially different from the crosshead separation rate.

In connection with the strain rate investigation, room-temperature creep tests were conducted on stress relieved and recrystallized specimens of unalloyed molybdenum. The creep data indicate that unalloyed molybdenum can exhibit measurable creep rates at room temperature at stresses below the yield strength.

The use of molybdenum as a structural engineering material has been appreciably extended in the past decade. The greater exploitation of this metal and its alloys has been predicated on its availability in large sizes in the form of bars, plates, sheets, and forgings. The increased application of the metal for structural purposes requires the establishment of standards and specifications for quality control and purchasing. Tensile properties of the metal become one of the items to appear in most specifications; therefore the procedures for performing the tension test must be standardized so that the results can be compared on a common basis.

Like other body-centered cubic metals, molybdenum is strain-rate sensitive. Bechtold,[2] Pugh,[3] Carreker and Guard,[4] and Alers and coworkers[5] have shown relatively wide variations in strength and ductility values with wide variations in rate of strain. Even at rates in the range normally used for tension testing of most metals, the yield strength of molybdenum will vary with relatively small changes in the rate of strain.

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* Presented at the Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] Climax Molybdenum Co. of Michigan, Detroit, Mich.

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It has been shown in the course of this investigation that the strain rate during tension testing must be monitored directly from the gage length of the tension specimen. Measurements of strain rate based on "crosshead separation," presetting of the valve controlling crosshead movement, or monitoring the rate of stress result in different gage-length strain rates, thus affecting the reported yield strength.

In view of the strain-rate sensitivity of molybdenum as well as the existence of strain aging, it would be expected that this metal might display measurable creep rates at room temperature. As part of this investigation, the creep rate at room temperature of unalloyed molybdenum was determined on specimens in the stress-relieved as well as in the fully recrystallized structural conditions.

TEST PROCEDURES

Bar stock rolled to $\frac{5}{8}$-in. or 2-in. diameter from vacuum arc-cast unalloyed molybdenum was available for the test program. Test specimen blanks were subjected to either a stress relief treatment or a recrystallization treatment prior to machining. Details of the thermal treatments are tabulated below:

Bar Diameter, in.	Stress Relief Treatment	Recrystallization Treatment
⅝	½ hr at 1800 F	1 hr at 2000 F
2	1 hr at 1800 F	1 hr at 2100 F

Test specimens were machined to a configuration consistent with ASTM Methods E 8.[6] Test-specimen reduced sections were $\frac{1}{4}$ in. in diameter by $1\frac{1}{4}$ in. long. Final elongation values were based on 1-in. gage lengths. Test specimens representing the 2-in. diameter bar stock were taken at a mid-radius location.

Elastic strain rates were monitored by means of microformer-type strain gages attached directly to the gage sections. After sufficient total deformation had occurred to permit calculation of a 0.2 per cent offset yield strength, the gage was removed (without stopping the crosshead motion) and the machine adjusted smoothly to achieve the desired plastic strain rate. The latter was measured by crosshead travel.

All tension tests were performed at room temperature (72 to 76 F). Specimens machined from the $\frac{5}{8}$-in. bar stock were tested at each of the strain rate combinations listed in Table I.

TABLE I.—STRAIN RATE COMBINATIONS USED IN TEST PROGRAM.

Elastic Rate, in per in. per min	Plastic Rate, in. per in. per min
0.0005	0.01
0.002	0.02
0.005	0.01
0.005	0.02
0.005	0.05
0.005	0.10
0.05	0.02

Two of the strain-rate combinations included in Table I were not applied to test specimens from the 2-in. bar stock. Duplicate tests were performed for a majority of the listed strain rate combinations.

Three tests were performed for the purpose of examining the differences between crosshead separation and actual strain rates. Stress-relieved specimens from the $\frac{5}{8}$-in. diameter stock were utilized. In the first of these tests, a specimen was strained elastically and through yielding at a rate of 0.0005 in. per in. per min, the rate being paced accurately from the signal from the microformer strain gage. At the same time the machine crosshead separation rate was measured by means of a dial indicator. In both the second and third tests, the crosshead separation rate was maintained constant (at 0.005 and 0.05

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[6] Tentative Methods of Tension Testing of Metallic Materials (E 8 - 57 T), 1958 Book of ASTM Standards, Part 3.

TABLE II.—ROOM-TEMPERATURE TENSION TEST RESULTS.

Strain Rate, in. per in. per min		Yield Strength, psi			Ultimate Strength, psi	Elongation in 1 in., per cent	Reduction of Area, per cent	Curve Type[a]
Elastic	Plastic	0.1 per cent	0.2 per cent	Drop in Load				
⅝-in. Diameter, Stress Relieved ½ hr at 1800 F								
0.0005	0.01	82 000	86 500	...	97 400	47	68.1	A
0.0005	0.01	80 700	84 800	...	98 500	41	70.5	A
0.0005[b]	0.01	80 900	82 200	...	97 300	47	71.0	A
0.002	0.02	87 600	89 000	...	101 900	41	68.5	A
0.005	0.01	...	...	95 200	96 500	46	67.6	B
0.005	0.01	93 700	95 300	...	97 500	41	67.8	A
0.005 CSR[c,d]	0.01	84 200	89 200	...	94 100	45	70.7	A
0.005	0.02	94 200	94 500	...	95 500	39	69.6	A
0.005	0.02	...	...	103 900	103 900	46	70.1	C
0.005	0.05	94 100	96 200	...	100 700	42	66.3	A
0.005	0.10	90 800	92 900	...	102 000	36	50.9	A
0.005	0.10	92 800	95 500	...	101 000	39	65.9	A
0.05	0.02	...	...	111 400	111 400	45	66.8	D
0.05	0.02	...	...	114 900	114 900	44	70.5	D
0.05 CSR[c,e]	0.01	95 600	99 100	...	104 000	43	70.7	A
⅝-in. Diameter, Recrystallized 1 hr at 2000 F								
0.0005	0.01	...	...	68 700	68 700	58	31.0	C
0.0005	0.01	...	...	68 200	68 200	44	26.0	C
0.002	0.02	...	...	71 900	74 300	48	38.0	B
0.005	0.01	...	...	87 800	87 800	53	54.6	D
0.005	0.01	...	...	87 600	87 600	55	44.6	D
0.005	0.02	...	...	84 200	84 200	46	37.8	D
0.005	0.02	...	...	82 700	82 700	56	57.2	D
0.005	0.05	...	...	82 900	82 900	47	37.9	D
0.005	0.10	...	...	87 200	87 200	34	28.1	D
0.005	0.10	...	...	85 500	85 500	40	35.4	D
0.05	0.02	...	...	93 700	93 700	46	30.7	D
0.05	0.02	...	...	101 900	101 900	59	57.4	D
2-in. Diameter, Stress Relieved 1 hr at 1800 F								
0.0005	0.01	...	...	74 200	81 100	48	73.8	B
0.0005	0.01	...	...	71 500	79 200	41	42.6	B
0.005	0.01	...	...	94 300	94 300	50	71.7	C
0.005	0.01	...	...	86 800	86 800	48	71.1	C
0.005	0.02	...	...	84 700	84 700	48	72.9	C
0.005	0.02	...	...	86 700	86 700	45	63.4	C
0.005	0.10	...	...	91 600	91 600	44	56.4	C
0.005	0.10	...	...	88 700	88 700	49	67.1	C
0.05	0.02	...	...	108 800	108 800	53	71.7	D
0.05	0.02	...	...	102 300	102 300	30	26.7	D
2-in. Diameter, Recrystallized 1 hr at 2100 F								
0.0005	0.01	...	...	62 900	65 700	41	33.6	B
0.0005	0.01	...	...	61 000	65 300	34	26.7	B
0.005	0.01	...	...	71 900	71 900	28	24.0	D
0.005	0.01	...	...	78 200	78 200	33	27.5	D
0.005	0.02	...	...	69 300	69 300	33	26.4	D
0.005	0.02	...	...	71 400	71 400	30	27.5	D
0.005	0.10	...	...	69 200	75 200	17	16.2	B
0.005	0.10	...	...	67 500	73 000	22	20.1	B
0.05	0.02	...	...	84 800	84 800	29	24.1	D
0.05	0.02	...	...	89 300	89 300	33	29.0	D

[a] See Fig. 1.

[b] See Fig. 3(*a*).

[c] CSR = crosshead separation rate.

[d] See Fig. 3(*b*).

[e] See Fig. 3(*c*).

in. per in. per min, respectively), while the actual rate at which the specimen was being strained was measured by the microformer gage. Crosshead separation rates were based on the specimen reduced section lengths. The plastic range in all three tests was traversed at 0.01 in. per in. per min.

Six room-temperature creep tests were performed on test specimens from the $\frac{5}{8}$-in. rolled bar stock. Three test specimens were machined from stress-relieved blanks and three from recrystallized stock. Test specimen dimensions were identical with those described above. Test specimens were fitted with resistance strain gages of the bakelite type. Specimens were loaded in conventional lever-type creep machines, and strain measurements were made at convenient time intervals.

TEST RESULTS

Tension test results are given in Table II. Four basic types of stress-strain curves were observed. Idealized versions of the four types have been drawn in Fig. 1 and cross-referenced in Table II. Curves *C* and *D* were quite similar, except that curve *D* was characterized by an upper yield point close to the elastic limit and a subsequent rapid drop in load. Curves of the *C* type, however, attained an upper yield point only after considerable plastic deformation. In curves of both the *C* and *D* types, the maximum stress after the rate change was lower than the upper yield stress. By convention, therefore, the ultimate strength and drop in load yield strength have the same value when behavior of this type is observed. Behavior of this type should not, of course, be confused with tests for which yield and ultimate strength values are similar by virtue of the absence of ductility.

It should be pointed out that the curves shown in Fig. 1 represent basic "types" only. It is possible, therefore, that curves for duplicate tests, although possibly falling into different categories, were nonetheless similar. In the case of duplicate tests on stress relieved specimens from the $\frac{5}{8}$-in. bar tested at rates of 0.005 and 0.01 in. per in. per min, the actual stress-strain curves were similar. The first listed, however, showed an extremely slight drop in load, whereas the first portion of the curve for the second listed test continued to rise slightly prior to the rate change.

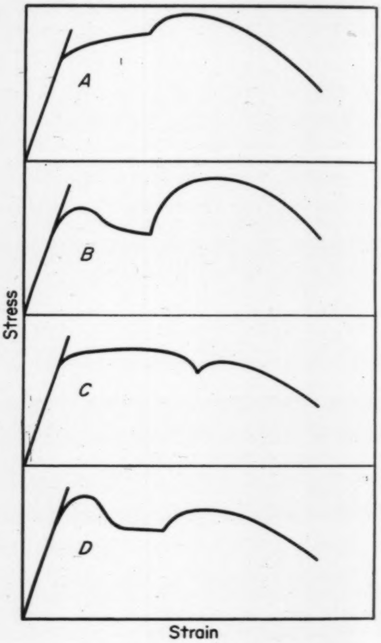


FIG. 1.—Stress-Strain Curve Configurations.

Elongation values were within the range from 17 to 59 per cent. With one exception, ductility values could not be correlated with strain rates. The exception was that recrystallized specimens from the 2-in. bar tested at the highest plastic rate (0.10 in. per in. per min) exhibited somewhat lower ductilities than the balance of the recrystallized specimens from this bar.

As one would expect, yield strength values tended to increase with increasing elastic strain rates. Yield strength values obtained for the $\frac{5}{8}$-in. bar stock are plotted against strain rate in Fig. 2. Average curves are drawn for 0.2 per cent offset yield strengths for stress relieved specimens and drop-in-load yield strengths for recrystallized specimens. Data points representing two tests conducted at constant crosshead separation rates are plotted at the actual strain rates effective at the time the yield strengths were measured. At relatively low elastic strain rates recrystallized molybdenum is significantly more rate-sensitive than stress relieved molybdenum. The average curves representing each structural condition tend to become parallel at the higher strain rates.

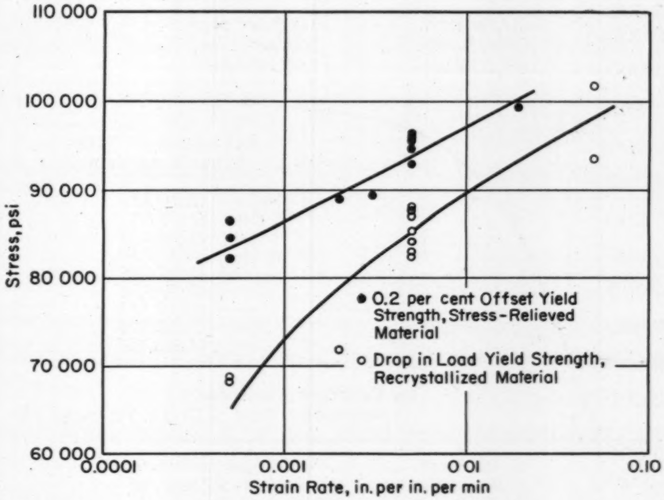
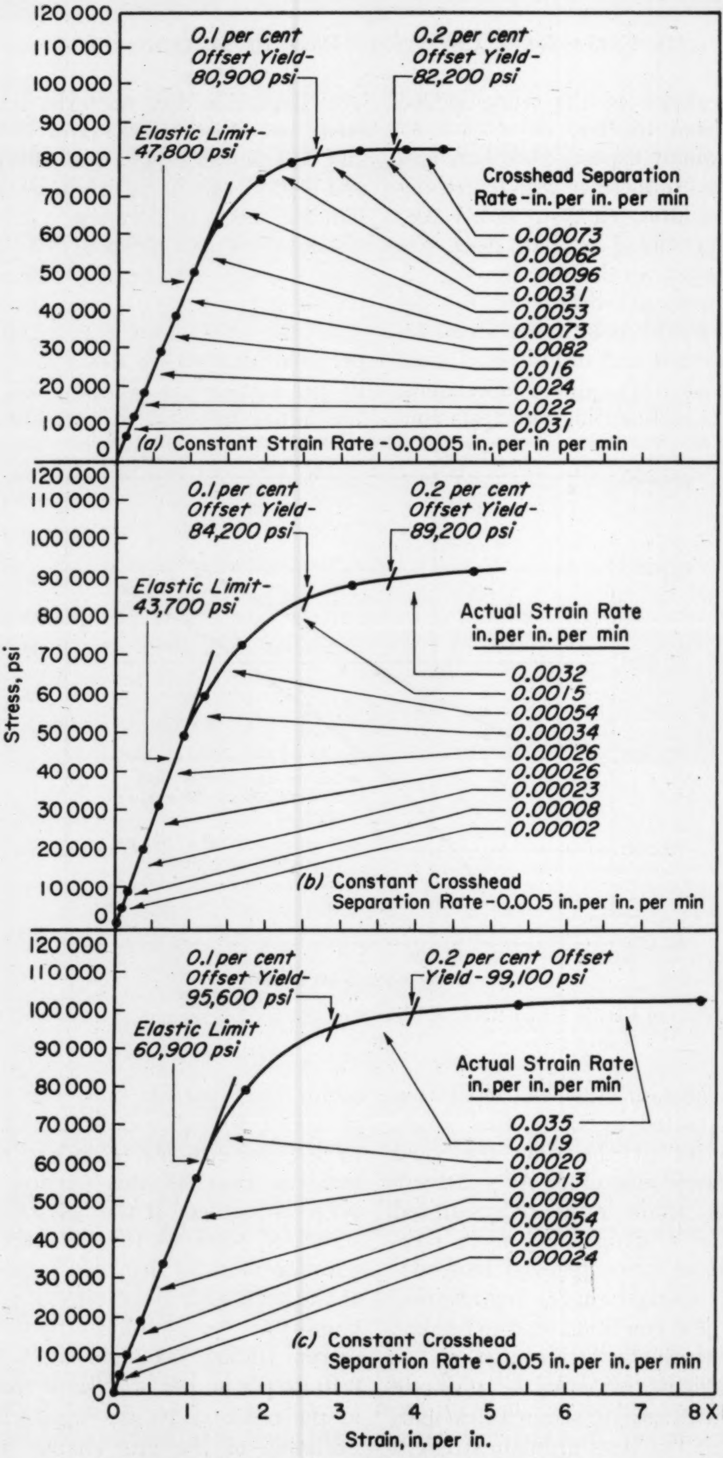


Fig. 2.—Effect of Elastic Strain Rate on Yield Strength of Molybdenum ($\frac{5}{8}$-in. Diameter Rolled Bar).

It is difficult to correlate ultimate strengths with plastic strain rates since for many of the tests ultimate strength and yield strength values were identical. This limitation does not exist for specimens tested at strain rates of 0.0005 and 0.01 in. per in. per min and of 0.005 and 0.10 in. per in. per min. These data indicate that an average increase in ultimate strength of about 8.4 per cent results from the tenfold increase in plastic strain rate.

In the opinion of the authors, the proposal of a plastic strain rate of 0.10 in. per in. per min is impractical. When the initial rate (called here the elastic strain rate) portion of a test is completed, it is highly desirable to adjust to and stabilize the secondary rate before the peak in this portion of the curve is reached. If the fast rate is not stabilized prior to this juncture, some question must be attached to the value at the peak load. In practice, it is almost impossible to stabilize hydraulically driven testing machines at a rate of 0.10 in. per in. per min, since something in the order of 10 sec elapses between initiation of the rate change and the peak in the plastic deformation curve.



(*a*) Constant strain rate of 0.0005 in. per in. per min.
(*b*) Constant crosshead separation rate 0.005 in. per in. per min.
(*c*) Constant crosshead separation rate 0.05 in. per in. per min.

FIG. 3.—Stress-Strain Curve for Room-Temperature Tension Test ($\frac{5}{8}$-in. Diameter Unalloyed Molybdenum Bar, Stress Relieved).

Some general commentary on the subject of tension testing of molybdenum is thought apropos. First, it is not possible to conduct accurately paced tension tests on molybdenum using mechanically driven (as opposed to hydraulically driven) testing machines. The reason is that in maintaining an accurate rate through yielding (for specimens exhibiting an upper yield point) it is necessary to have infinitely variable crosshead speed control down to zero. Because of inertial effects in mechanically driven machines, they have a finite low speed limit and cannot be operated between this speed and zero. Second, it is not possible to base strain rates on crosshead motion during the early stages of testing. It is suspected that many of the discrepancies in data from various sources result from one or both of the above limitations.

TABLE III.—ROOM-TEMPERATURE TENSION TEST RESULTS FOR $\frac{1}{16}$-IN. UNALLOYED MOLYBDENUM SHEET STRESS RELIEVED.

Strain Rate, in. per in. per min		Yield Strength, psi		Ultimate Strength, psi	Elongation, per cent	Curve Type[a]
Elastic	Plastic	0.1 per cent	0.2 per cent			
0.005[b]	0.02	92 500	93 000	105 000	30	A
0.005 CSR[c,d]	0.02	86 000	89 100	105 900	30	A

[a] See Fig. 1.
[b] See Fig. 4(*a*).
[c] CSR = crosshead separation rate.
[d] See Fig. 4(*b*).

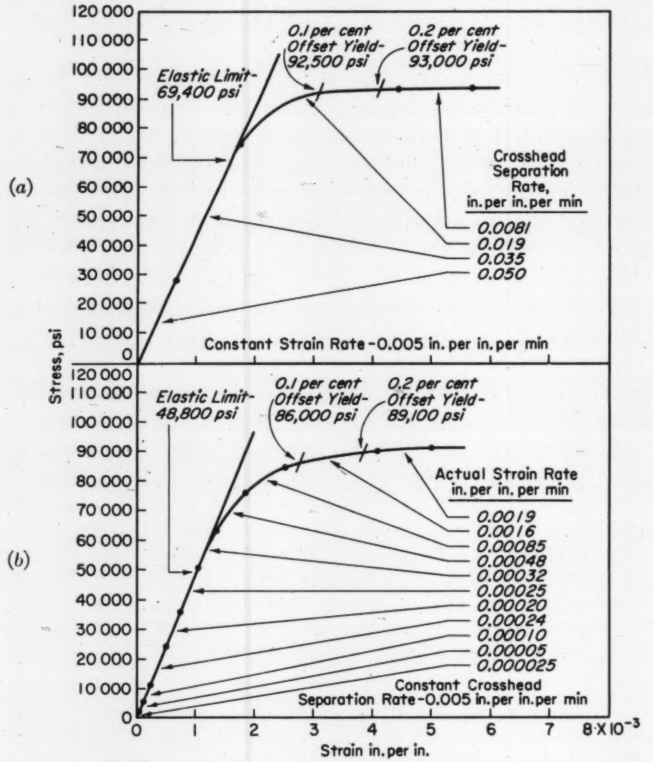
The results of tests performed to demonstrate the latter point, that is, the discrepancy between crosshead separation rate and actual strain rate, are given in Table II and plotted in Fig. 3.

The first test (Fig. 3(*a*)) was conducted at an actual strain rate of 0.0005 in. per in. per min as monitored by the microformer strain gage. Concomitant crosshead separation rates are noted on the curve. The indicated crosshead rates are average values for the strain increments between adjacent closed circles. It is evident from this test that the crosshead separation rate bore no similarity to the actual strain rate until significant plastic deformation had occurred. In the early stages of elastic deformation, the crosshead separation rate was quite high. In the vicinity of the elastic limit, it was necessary to reduce the crosshead speed in order to maintain the predetermined specimen strain rate.

During the second test (Fig. 3(*b*)) the crosshead separation rate was maintained at 0.005 in. per in. per min. The actual strain rate occurring in the specimen was measured by the microformer strain gage. Over a hundredfold increase in actual strain rate was observed during this test. Again, only after substantial plastic strain occurred did the actual strain rate increase sufficiently to approach the selected crosshead separation rate.

The third test (Fig. 3 (*c*) was conducted in a manner similar to the second except that the crosshead separation rate was 0.05 in. per in. per min. Again the actual strain rate increased during the test, and at the conclusion of the test was over 100 times that at the initiation of loading.

Two tension tests were performed on unalloyed molybdenum sheet ($\frac{1}{16}$ in. thick) in order to introduce the additional variable of specimen configuration.



(*a*) Constant strain rate 0.005 in. per in. per min.
(*b*) Constant crosshead separation rate 0.005 in. per in. per min.

FIG. 4.—Stress-Strain Curve for Room-Temperature Tension Test ($\frac{1}{16}$-in. Thick Unalloyed Molybdenum Sheet, Stress Relieved).

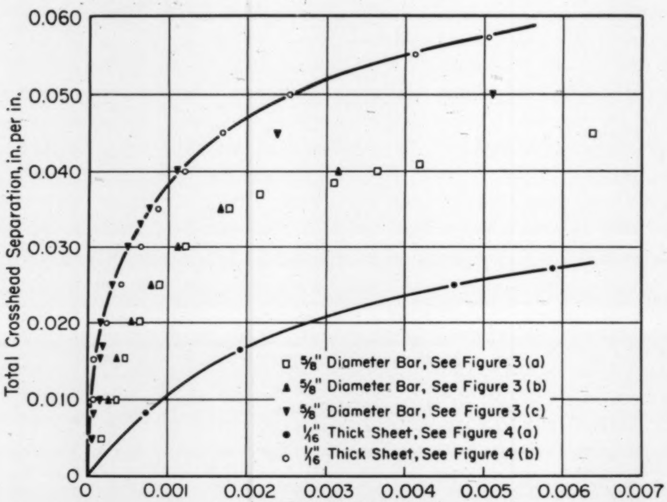


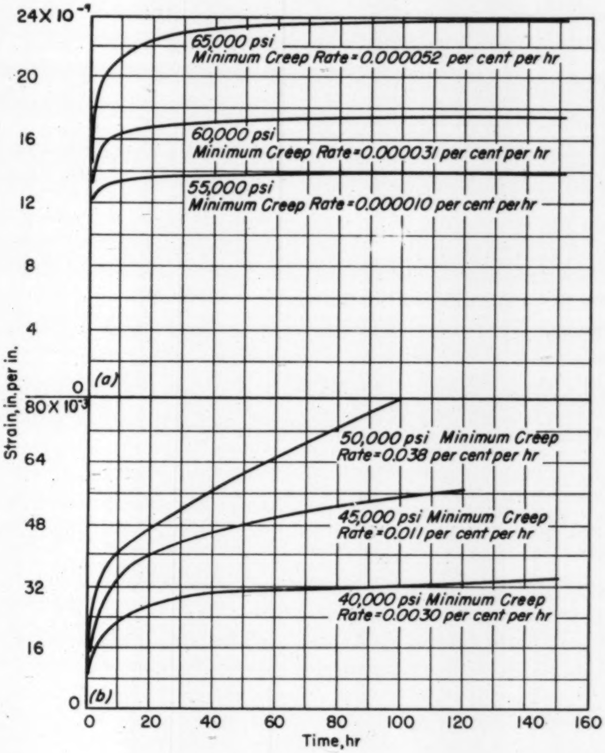
FIG. 5.—Relationship Between Actual Strain and Crosshead Separation During Tension Testing of Unalloyed Molybdenum.

Tension test specimens were machined with reduced sections $\frac{1}{2}$ in. in width by $2\frac{3}{8}$ in. in length. Test specimen blanks were given a stress-relief treatment consisting of 15 min at 1800 F.

Test results are given in Table III. The initial portions of each stress-strain curve are plotted in Fig 4(*a*) and (*b*).

The disparity between crosshead separation rate and specimen strain rate is again evident from these data. It is significant that differences in strain rates were observed for tests conducted under identical crosshead separation rates but involving different specimen configurations (cylindrical specimen, Fig. 3(*b*), and rectangular cross-section specimen, Fig. 4(*b*)).

The disparity between total strain and total crosshead separation is shown in Fig. 5. The results of three tests on cylindrical specimens and two tests on sheet specimens define a broad band in the relationship described in Fig. 5. It is concluded that the difference between strain rate and crosshead separation rate during the early stages of tension testing is not only sensitive to specimen geometry but is in itself rate-sensitive.



(*a*) Stress relieved.
(*b*) Fully recrystallized.

FIG. 6.—Room-Temperature Creep Test Results for Unalloyed Molybdenum ($\frac{5}{8}$-in. Rolled Bar Stock).

It should be evident from the data presented that the actual strain rate during the early stages of deformation must be precisely controlled if accurate, reproducible results are to be obtained.

It has been proposed that if molybdenum is rate-sensitive at room temperature (as it certainly is), it probably exhibits finite creep rates at room temperature. In order to characterize the creep behavior of unalloyed molybdenum at room temperature, a series of creep tests was conducted. Results of room-temperature creep tests are presented in Fig. 6(*a*) and (*b*). Tests on both stress-relieved and recrystallized specimens revealed relatively high creep rates during the first few hours under load. After 20 to 30 hr, however, the creep rate had diminished significantly.

Although stress levels were lower for the recrystallized specimens, minimum creep rates were several orders of magnitude higher than for stress relieved specimens.

Average elastic limit values measured for replicate tests conducted on $\frac{5}{8}$-in. bars at elastic strain rates of 0.0005 in. per in. per min were: stress-relieved, 51,300 psi; and recrystallized, 45,900 psi. It is evident that room-temperature creep behavior cannot be correlated with elastic limit.

Stress-relieved test specimens loaded above the elastic limit displayed low creep rates, while much higher rates were measured for two recrystallized specimens loaded at stresses below the appropriate elastic limit value.

The data developed during the course of this investigation, as well as those reported by other investigators, indicated that strain aging is an important factor in the mechanical behavior of molybdenum. Tests on fully recrystallized specimens consistently displayed a yield point discontinuity whereas tests on stress-relieved specimens of the $\frac{5}{8}$-in. diameter stock tended toward a continuous type of yielding. This would indicate that strain aging, a diffusion of interstitially dissolved atoms to dislocation sites and consequent pinning of dislocations, is occurring during straining of the work-hardened (stress-relieved in this case) material. Interestingly, two tests performed on stress-relieved specimens using a very high initial strain rate (0.05 in. per in. per min) displayed yield point discontinuities. The explanation of this behavior is that the rate of strain applied to the specimens was high enough to cause the dislocations to break away from their locking atmospheres of interstitial solute atoms, and their rate of movement was sufficiently greater than the diffusion rate of the solute atoms to prevent subsequent interaction.

Since a reaction of the strain-aging type evidently does not contribute to the strength of fully recrystallized molybdenum, it is suggested that the low room-temperature creep rates assigned to stress-relieved molybdenum are attributable to the strain aging phenomenon.

SUMMARY

The strain-rate sensitivity of molybdenum has been verified. Yield strengths were found sensitive to elastic rates and tensile strengths sensitive to plastic rates. Ductility was not particularly sensitive over the range of strain rates studied here. Minimum creep rates for recrystallized specimens were several orders of magnitude higher than those for stress-relieved specimens. Strain aging was concluded to be an important factor in the mechanical behavior of molybdenum.

TENSILE STRENGTH - HARDNESS CORRELATION FOR TITANIUM ALLOYS*,†

By Charles F. Hickey, Jr.[1]

Synopsis

This investigation was concerned with the establishment of a correlation between tensile strength and hardness for titanium alloys. Materials of current interest were considered and tested over a fairly wide property range. Tensile and hardness properties were obtained as a function of testing temperature, heat treatment, and section size of material. Data were also obtained from the literature.

As a result of the findings, a correlation was established between tensile strength and hardness for stable alpha-beta alloys. It does not apply for metastable alpha-beta alloys, nor does it adequately define data in all-beta alloys. In addition, a hardness correlation was established between Vickers and Rockwell C readings for the stable alpha-beta alloys.

The tensile strength - hardness relationship provides a fast, inexpensive means of approximating tensile strength from a simple hardness test. Such a relationship exists in steel (**1,2**),[2] where the ultimate tensile strength is approximately 500 times the Brinell hardness number. Attempts have been made in the past to establish a workable correlation for titanium, but for the most part this has met with little success since hardness apparently varies with alloy type, directionality, prior fabrication history of the alloy, and the phases present in the alloy. Thus it appears that a strength-hardness correlation for titanium might vary considerably from one alloy to another and even with different processing variables for a given alloy.

Realizing the above, it was the author's opinion that sufficient work had not been done on the subject to eliminate completely the possibility that a correlation may exist, at least within certain alloys.

This report is a presentation of tensile strength - hardness data for numerous titanium alloys of current interest and importance. Data for the program have been accrued from tests conducted at Watertown Arsenal Laboratories, previously published work by the author (**3**), and from a literature survey (**4,5**).

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* Presented at the Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

† The statements and opinions expressed in this article are those of the author and do not necessarily indicate the views or policy of the Army Ordnance Corps.

[1] Watertown Arsenal Laboratories, Watertown, Mass.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

MATERIALS AND PROCEDURE

Materials selected for investigation were Department of Defense titanium alloys and others of commercial and current interest. The designated alloys, with all available compositions, are given in Table I.

Hardness data obtained from the literature were reported in Rockwell C (R_c) units for some alloys, and Vickers hardness number (VHN), frequently referred to as diamond pyramid hardness (DPH), for others. Thus, for the alloys which were to be investigated at Watertown Arsenal Laboratories it was decided to utilize both hardness methods in hopes of also establishing a relationship between R_c and VHN readings. This would allow greater utilization of literature data and would also allow a tensile strength - hardness relationship to be expressed in terms of either R_c or VHN hardness.

Tensile and hardness properties were obtained as a function of testing temperature, heat treatment, and section size of the material. Standard 0.252-in. tension specimens were used in all cases. R_c data were obtained from Charpy bars, while VHN data were obtained from polished disks. The R_c data are from a plane perpendicular to the tensile direction while VHN data are parallel to the tensile direction. Hardness direction was not stipulated in the literature data.

The least mean square method was used for fitting equations to the various curves. It was assumed that a linear relationship would define each plot.

TABLE I.—COMPOSITION OF ALLOYS INVESTIGATED.

Titanium Alloy	Element, per cent by weight								
	Aluminum	Molybdenum	Vanadium	Chromium	Hydrogen	Oxygen	Carbon	Nitrogen	Iron
7Al-4Mo	7.16	4.19	...	...	0.0036	0.08	0.065	0.007	...
7Al-3Mo	7.15	3.00	...	...	0.0126	0.09	0.040	0.009	0.20
6Al-4V	5.90	...	4.25	...	0.0012	0.09	0.030	0.010	...
4Al-3Mo-1V	3.85	3.07	0.96	...	0.0024	0.06	0.045	0.009	...
16V-2.5Al	2.79	...	15.80	...	0.0018	0.09	0.025	0.025	...
13V-11Cr-3Al	3.18	...	13.15	10.96	0.0069	0.09	0.028	0.013	...
6Mo-3Al[3]	2.77	6.00	...	...	0.0024	0.13	0.024	0.018	0.02
6.5Al-3Mo-1V[3]	6.39	2.92	1.00	...	0.0044	0.11	0.028	0.015	0.02
6Al-4V[3]	5.85	...	4.07	...	0.0042	0.11	0.026	0.018	0.162
4Al-3Mo-1V[4]	...	...	...	...	...	...	...	...	...
6Al-4V[5]	...	...	...	...	...	...	...	...	...

RESULTS AND DISCUSSION

Effect of Testing Temperature on Hardness:

Figure 1 is a plot of Vickers hardness versus testing temperature for Ti-7Al-4Mo, Ti-7Al-3Mo, Ti-6Al-4V, Ti-4Al-3Mo-1V, Ti-16V-2.5Al, and the all-beta alloy Ti-13V-11Cr-3Al in the annealed condition. All-beta Ti-13V-11Cr-3Al shows the greatest increase in hardness over the investigated temperature span. It ranges from approximately 269 VHN at 200 F to 544 VHN at −319 F, a difference of 275. The five remaining alloys follow the same general trend with an increase of between 180 and 220 VHN. Ti-16V-2.5Al is of a much lower hardness level than any of the other materials; however, its increase with a decrease in temperature is within the above stated range.

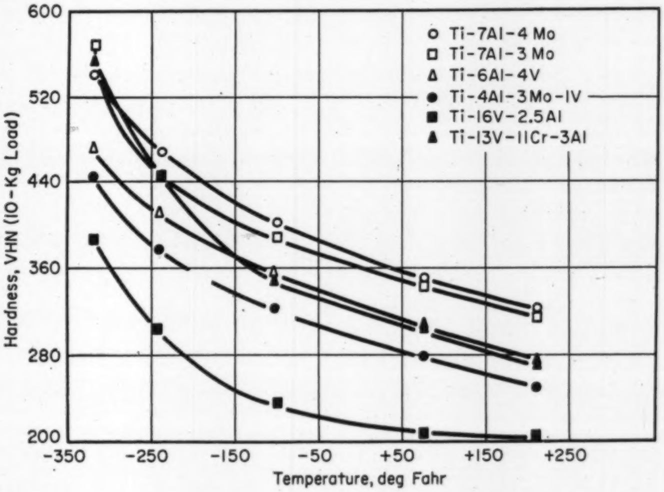


FIG. 1.—Effect of Testing Temperature on Hardness of Titanium Alloys.

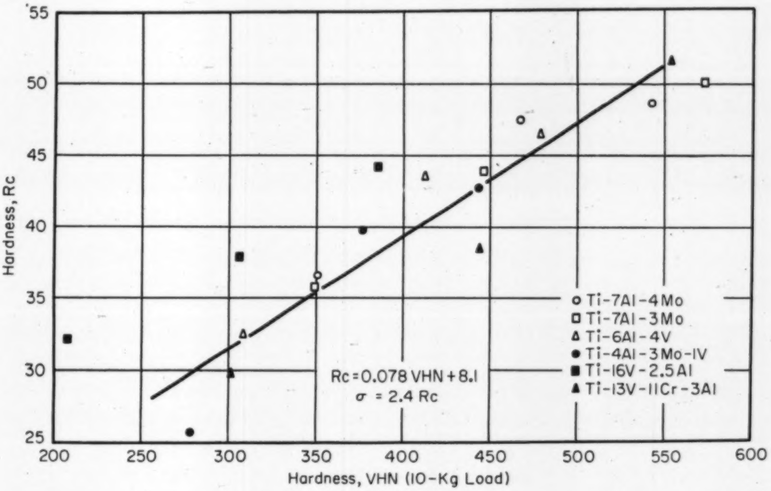


FIG. 2.—Effect of Testing Temperature (Room Temperature to −319 F) on Hardness Correlation (R_c Versus VHN) for Titanium Alloys.

Hardness Correlation:

Figure 2 is a plot of R_c versus VHN readings for the alloys in Fig. 1, utilizing testing temperatures of room temperature, −240, and −319 F. An examination of the data shows the hardness level of Ti-16V-2.5Al and all-beta Ti-13V-11Cr-3Al to be higher and lower, respectively, than that of the other alloys; thus they were not included in the relationship which is defined by the following equation:

$$R_c = 0.078 \text{ VHN} + 8.1$$

$$\sigma = 2.4 \ R_c$$

A standard deviation of 2.4 R_c means

that 68 per cent of the cases (data points) were included within the range of ±2.4 R_c about the line of regression which is defined by the equation $R_c = 0.078\ \text{VHN} + 8.1$.

The reason for the higher hardness level of metastable Ti-16V-2.5 Al is contained in the fact that the beta phase is mechanically unstable and transforms to martensite upon straining. In the Vickers test, a load of 10 kg is used while the Rockwell C employs a load of 150 kg. Therefore, because of the greater load in the latter test, more strain is imparted, thus accounting for the higher hardness values. Verification of this is shown in Fig. 3 which shows that as the Vickers load is increased to 50 kg the resulting hardness becomes greater for the metastable alloy. It is also shown utilizing Ti-7Al-4Mo, that hardness is independent of load for stable alpha beta alloys.

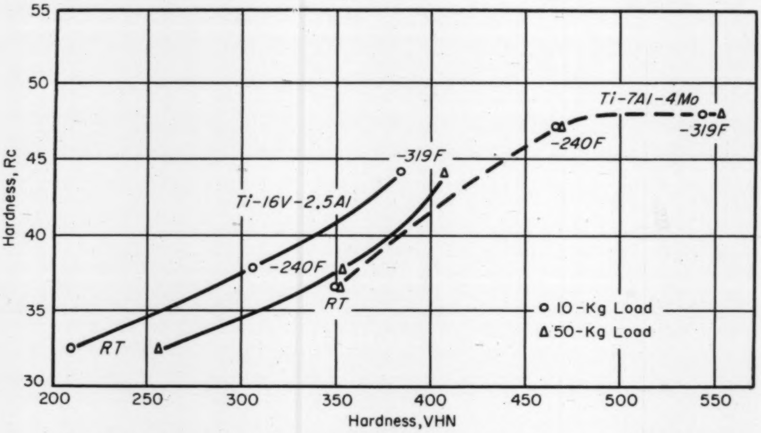


FIG. 3.—Effect of Load on Hardness Relationship (VHN Versus R_c) for Titanium Alloys.

For reasons to be discussed later, all-beta Ti-13V-11Cr-3Al data were not included in the hardness equation.

Tensile Strength Versus Hardness Relationships:

Figures 4(*a*) and (*b*) are plots of tensile strength versus hardness, VHN and R_c respectively, of the afore-mentioned materials with data being obtained at room temperature, −105, −240, and −319 F for most of the alloys. In both figures the four stable alpha-beta alloys exhibit a similar trend which can be defined as follows:

$$\text{Ultimate tensile strength} = 435\ \text{VHN} + 9200$$

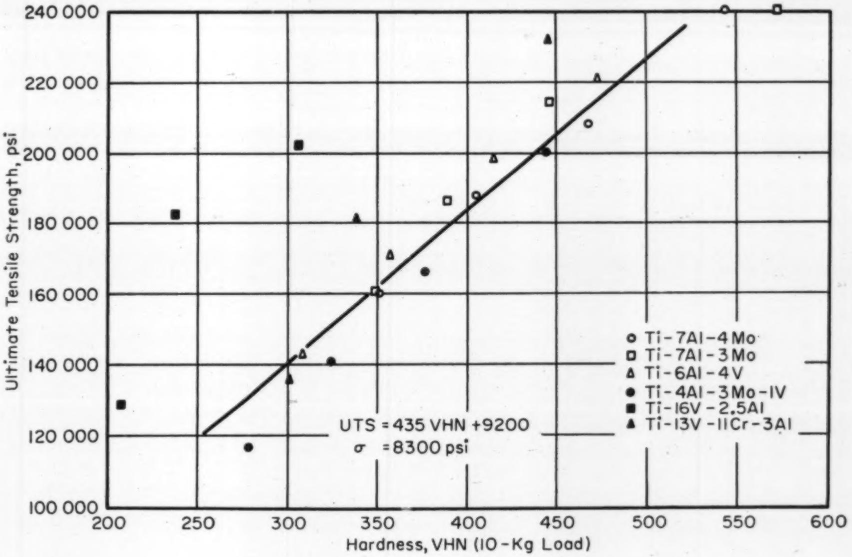
$$\sigma = 8300\ \text{psi}$$

$$\text{Ultimate tensile strength} = 5200\ R_c - 24{,}000$$

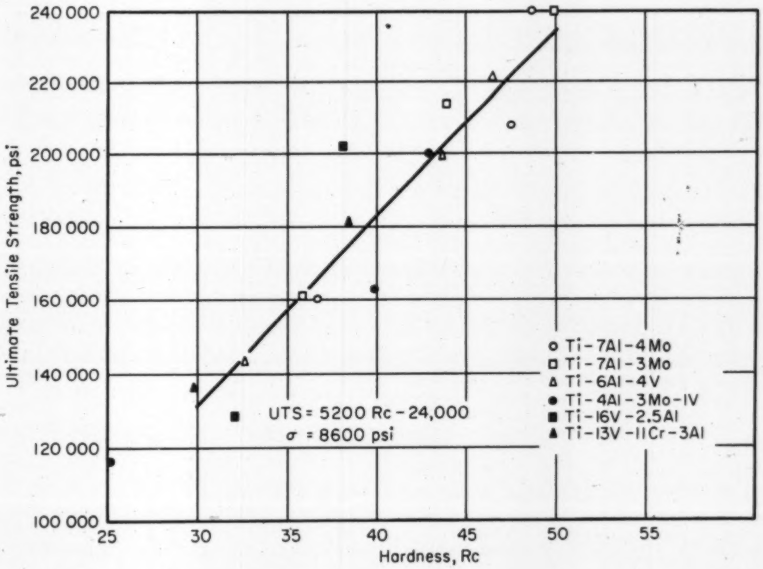
$$\sigma = 8600\ \text{psi}$$

Again, as in the hardness correlation, the Ti-16V-2.5Al data fell above that of other alloys. Strain-induced martensitic transformation once more appears to be the answer. The strain produced by the tension test is considerably higher than that of either hardness method, accounting for the high corresponding tensile values.

It should be mentioned that data for the all-beta alloy Ti-13V-11Cr-3Al were not included in either equation, even though at some temperatures they agree fairly well with the stable alpha-beta alloys. In Fig. 4(*a*) there is some evidence that the alloy may undergo a certain amount of martensitic transfor-



(*a*) Vickers hardness number.



(*b*) Rockwell hardness, C scale.

FIG. 4.—Tensile Strength - Hardness Relationship as a Function of Testing Temperature (Room Temperature to −319 F) for Titanium Alloys.

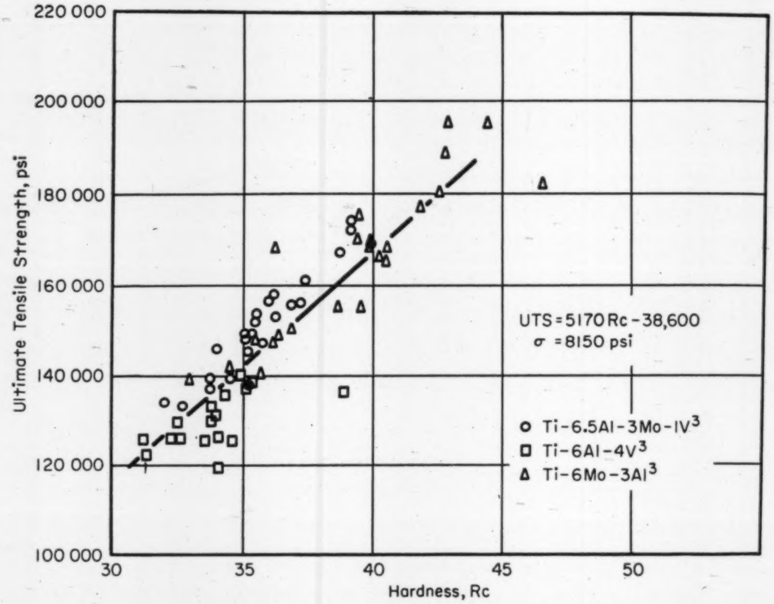


Fig. 5.—Tensile Strength - Hardness Relationship as a Function of Heat Treatment and Section Size for Titanium Alloys.

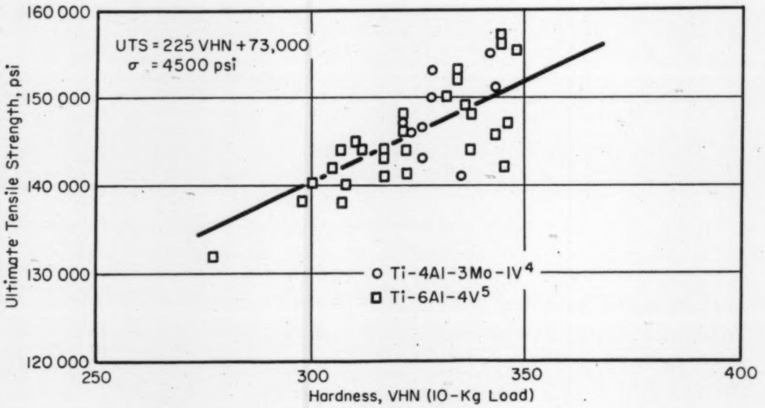


Fig. 6.—Tensile Strength - Hardness Relationship for Titanium Alloys.

mation due to straining; also, it is known that this material exhibits excessive embrittlement at cryogenic temperatures.

Figure 5 is a plot of tensile strength versus hardness (R_c) utilizing tensile data which has been previously published (3), and the corresponding hardness data. The materials, Ti-6Al-4V, Ti-6.5Al-3Mo-1V, and Ti-6Mo-3Al are stable alpha-beta alloys. Each material was of several section diameters (2, 3, 4, and 6 in.) and tested in both the annealed and heat-treated conditions, thus providing a range of conditions and properties.

The data fell into a fairly consistent

pattern and can be described as follows:

$$\text{Ultimate tensile strength} = 5170\ R_c - 38{,}600$$

$$\sigma = 8150 \text{ psi}$$

Figure 6 is a plot of ultimate tensile strength versus VHN for Ti-4Al-3Mo-1V[4] and Ti-6Al-4V[5], the data having been obtained from Battelle Memorial Inst. The first alloy was tested in the heat-treated condition and the latter is cast material which presumably was annealed prior to testing. Their data may be defined as follows:

$$\text{Ultimate tensile strength} = 225\ \text{VHN} + 73{,}000$$

$$\sigma = 4500 \text{ psi}$$

As can be seen in Fig. 6, the property range is very limited. Due to this fact, and lacking complete fabrication history, only limited emphasis can be placed upon these data. However, it can be said that in the area of 300 VHN fairly good agreement equation-wise does exist between these data and those presented in Fig. 4.

Figures 4 to 6 present tensile strength versus hardness data for numerous titanium alloys in various conditions. In an effort to arrive at the equation which best defined all the data, it was decided to analyze all the results on an R_c versus ultimate tensile strength basis. The VHN's in Fig. 6 were converted to R_c utilizing the hardness relationship established in Fig. 2. Also included in this bulk analysis were limited results from Ti-5.5Al-5.5V-2Sn, Ti-155A, and Ti-7Al-4V.

The equation and resulting deviation which best describe all of the data are:

$$\text{Ultimate tensile strength} = 5050\ R_c - 27{,}000$$

$$\sigma = 8250 \text{ psi}$$

Utilizing the hardness conversion between R_c and VHN, the hardness versus tensile strength relationship can now be

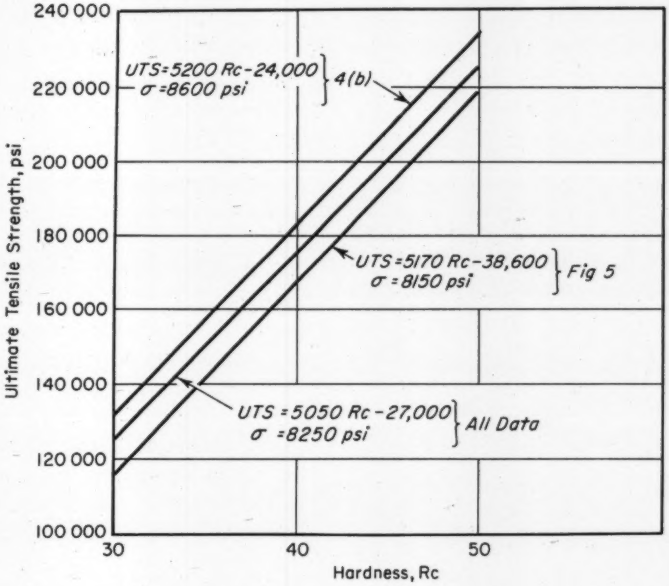


FIG. 7.—Summary of Tensile Strength - Hardness Relationship for Titanium Alloys.

expressed in terms of VHN, as follows:

$$\text{Ultimate tensile strength} = 395 \text{ VHN} + 14{,}000$$

Figure 7 is a plot of the equation which best defines the presented data. In addition there are also equation plots of the data presented in Figs. 4(*b*) and 5. It is of interest to note that the data from these two figures parallel each other, thus tending to indicate that the slope of the curve is independent of the materials testing condition. It can also be seen that the standard deviation for all of the data encompass these two plots at nearly all hardness levels.

After the statistical analysis had been completed for this paper, additional data on tensile strength and Vickers hardness for Ti-4Al-3Mo-1V and Ti-6Al-4V by Rice, Campbell, and Simmons (6) were discovered. Thus, these results could not be included; however it can be said that their findings fall within the scope of the correlation.

CONCLUSIONS

The results of this investigation to establish a correlation between tensile strength and hardness for titanium are as follows:

1. A hardness correlation (R_c versus VHN) was established for stable alpha-beta alloys, based upon the data from Ti-7Al-4Mo, Ti-7Al-3Mo, Ti-6Al-4V, and Ti-4Al-3Mo-1V. The equations and standard deviation are as follows:

$$R_c = 0.078 \text{ VHN} + 8.1$$
$$\sigma = 2.4 \; R_c$$

2. A relationship does exist for stable alpha-beta alloys over the investigated hardness range of 30 to 50 R_c and corresponding tensile strengths of 130,000 to 240,000 psi. This relationship and standard deviation can be expressed as follows:

$$\text{Ultimate tensile strength} = 5050 \; R_c - 27{,}000$$
$$\sigma = 8250 \text{ psi}$$

3. The above relationships can be combined, resulting in the following expression for ultimate tensile strength in terms of VHN:

$$\text{Ultimate tensile strength} = 395 \text{ VHN} + 14{,}000$$

This study shows that the tensile strength - hardness relationships for the alpha-beta materials does not define Ti-16V-2.5Al, a metastable alpha-beta alloy. A likely explanation appears to lie in the fact that this alloy transforms to martensite upon straining.

Even though the data for all-beta Ti-13V-11Cr-3Al agree fairly well with the stable alpha-beta results, especially at lower strength and hardness levels, they were not included in the above equations. This decision was based on the possibility that the alloy may undergo a certain amount of martensitic transformation due to straining, plus the known fact that it exhibits excessive embrittlement at cryogenic temperatures.

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MECHANICAL PROPERTIES OF TITANIUM ALLOYS AS A FUNCTION OF HEAT TREATMENT AND SECTION SIZE*

By Charles F. Hickey, Jr.[1]

Synopsis

The mechanical properties of 6Al-4V, 6.5Al-3Mo-1V, and 6Mo-3Al titanium alloys were investigated as a function of heat treatment and section size. Sections of 2-, 3-, 4-, and 6-in. diameter rounds were studied, each alloy being subjected to two solution temperatures followed by three aging temperatures. Tensile and impact data were obtained in all cases. As a result of the findings an optimum heat treatment was established for each alloy. Other factors such as specimen location, directionality, and microstructure are discussed.

Development of heat-treatment procedures for titanium alloys of all types has in the past been confined mainly to laboratory-scale operations. When these procedures are tried on a production basis, reported mechanical properties are not always realized. Much time and material has been lost in attempting without success to use these published data; then a temporary program must be set up to determine optimum practice for a single alloy or component.

A major problem often encountered is the variation in properties between different sizes of stock. This variation can arise from differences in microstructure brought about by variations in prior processing, or from differences in cooling rate as various sizes of stock are heat treated. Data on the effect of section size **(1–3)**[2] are limited and there have been few publications on that subject to date.

Materials and Procedures

The three titanium alloys investigated, 6Al-4V, 6.5Al-3Mo-1V, and 6Mo-3Al, were procured in the form of forged bar stock 2, 3, 4, and 6 in. in diamter. All diameters within each material were from the same heat. The 4-in. bar for the Ti-6Al-4V alloy could not be supplied as such, thus it was not available for testing. All materials were the product of a 20-in. minimum diameter ingot, double melted by the consumable electrode process from sponge of 120 to 140 Brinell hardness number.

The chemical analysis for each of the three alloys is given in Table I.

The heat treatments, shown in Table II, consisted of heating at a high or a low solution temperature and then water quenching, followed by 3 aging cycles per bar diameter. The material was received in the mill annealed condition and heat treated in the full section size prior to machining. The section lengths ranged from $8\frac{1}{4}$ in. for the 2-in. diameter bars to

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* The statements and opinions expressed in this article are those of the author and do not necessarily indicate the views or policy of the Army Ordnance Corps.

[1] Watertown Arsenal Laboratories, Watertown, Mass.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

$11\frac{3}{4}$ in. for the 6-in. diameter materials. The higher solution temperature for each alloy was approximately 100 F below the beta transus, and the lower value was the temperature at which the minimum yield-tensile ratio existed in the solution-treated condition.

Longitudinal and transverse tension and Charpy V notch specimens were machined from each diameter bar at both core and midwall location. Longitudinal Charpy specimens were notched perpendicular to the longitudinal direction, while transverse specimens were notched parallel to it. The side of the specimens on which the notch was located was toward the core of the original section.

Tension testing was conducted at room temperature at a strain rate of 0.005 in. per in. per min up to 0.2 per cent offset, followed by a platen speed of 0.02 in. per min to fracture. A 120,000-lb capacity Universal hydraulic machine was used for testing.

Charpy impact testing was performed at −40 C on a 240 ft-lb Sonntag machine.

RESULTS AND DISCUSSION

Effect of Heat Treatment:

The three titanium alloys investigated, 6Al-4V, 6.5Al-3Mo-1V, and 6Mo-3Al, are all α-β alloys. However, because of the difference which exists between their compositions, it is to be expected that the as-received annealed mechanical properties will be increased by varying amounts when heat treated. This increase, plus the change in properties within a particular section size for a given alloy over a range of heat treating temperatures, is a measure of the alloys' sensitivity to heat treatment.

Data show that the as-received

TABLE I.—CHEMICAL COMPOSITION OF ALLOYS TESTED.

Alloy	Composition, per cent by weight							
	Aluminum	Vanadium	Molybdenum	Carbon	Nitrogen	Iron	Hydrogen	Oxygen
Ti-6Al-4V	5.85	4.07	...	0.026	0.018	0.162	0.0042	0.108
Ti-6.5Al-3Mo-1V	6.39	1.00	2.92	0.028	0.015	0.02	0.0044	0.110
Ti-6Mo-3Al	2.77	...	6.00	0.024	0.018	0.02	0.0024	0.132

TABLE II.—HEAT TREATMENTS OF ALLOYS TESTED.

Alloy	Solution Treatment	Aging Treatment
Ti-6Al-4V	1550 F for 1 hr, water quench	950, 1050, and 1150 F for 16 hr, air cool
	1650 F for 1 hr, water quench	950, 1050, and 1150 F for 1 hr per in. of diameter, air cool
Ti-6.5Al-3Mo-1V	1650 F for 1 hr, water quench	1000, 1100, 1200 F for 24 hr, air cool
	1760 F for 1 hr, water quench	1000, 1100, 1200 F for 1 hr per in. of diameter, air cool
Ti-6Mo-3Al	1550 F for 1 hr, water quench	800, 900, 1000 F for 24 hr, air cool
	1625 F for 1 hr, water quench	800, 900, 1000 F for 1 hr per in. of diameter, air cool

TABLE III.—LONGITUDINAL AND TRANSVERSE CORE MECHANICAL PROPERTIES FOR Ti-6Al-4V ALLOY.[a]

Condition[b]	Property	2-in. Section			3-in. Section			6-in. Section		
		Longitudinal	Transverse	Δ	Longitudinal	Transverse	Δ	Longitudinal	Transverse	Δ
U	0.1 yield strength, psi	128 000	142 000	14.0	125 000	131 000	6.0	121 000	125 000	4.0
	Tensile strength, psi	139 000	144 000	5.0	134 000	139 000	5.0	133 000	134 000	1.0
	Elongation, per cent	15.4	11.6	−3.8	14.4	10.1	−4.3	16.0	11.4	−4.6
	Reduction of area, per cent	38.5	36.6	−1.9	37.3	31.5	−5.8	37.1	24.3	−12.8
	Impact, ft-lb	21.6	10.9	−10.7	24.5	13.4	−11.1	24.7	14.7	−10.0
1	0.1 yield strength, psi	137 000	150 000	13.0	133 000	139 000	6.0	125 000	132 000	7.0
	Tensile strength, psi	149 000	153 000	4.0	143 000	146 000	3.0	136 000	140 000	4.0
	Elongation, per cent	15.4	12.5	−2.9	14.7	10.9	−3.8	15.0	13.3	−1.7
	Reduction of area, per cent	38.4	44.0	5.6	36.4	28.4	−8.0	32.6	32.0	−0.6
	Impact, ft-lb	22.8	11.3	−11.5	25.1	13.0	−12.1	23.5	13.2	−10.3
2	0.1 yield strength, psi	135 000	145 000	10.0	131 000	137 000	6.0	126 000	130 000	4.0
	Tensile strength, psi	143 000	148 000	5.0	137 000	141 000	4.0	134 000	136 000	2.0
	Elongation, per cent	15.7	14.1	−1.6	15.3	10.9	−4.4	14.0	12.5	−1.5
	Reduction of area, per cent	39.0	44.8	5.8	36.2	27.9	−8.3	33.3	27.5	−5.8
	Impact, ft-lb	20.7	11.2	−9.5	23.6	11.6	−12.0	19.0	10.8	−8.2
3	0.1 yield strength, psi	130 000	141 000	11.0	126 000	133 000	7.0	122 000	126 000	4.0
	Tensile strength, psi	150 000	148 000	−2.0	142 000	141 000	−1.0	136 000	131 000	−5.0
	Elongation, per cent	16.0	12.5	−3.5	16.0	12.5	−3.5	15.7	14.0	−1.7
	Reduction of area, per cent	39.1	36.7	−2.4	36.5	31.5	−5.0	36.7	30.1	−6.6
	Impact, ft-lb	19.5	10.7	−8.8	21.6	12.3	−9.3	21.5	11.1	−10.4
4	0.1 yield strength, psi	126 000	144 000	18.0	125 000	129 000	4.0	118 000	124 000	6.0
	Tensile strength, psi	150 000	156 000	6.0	142 000	146 000	4.0	136 000	135 000	−1.0
	Elongation, per cent	15.0	14.1	−0.9	15.0	11.8	−3.2	15.7	11.7	−4.0
	Reduction of area, per cent	44.8	45.3	0.5	39.0	31.5	−7.5	39.4	35.2	−4.2
	Impact, ft-lb	26.3	13.8	−12.5	32.1	17.1	−15.0	28.4	17.0	−11.4
5	0.1 yield strength, psi	140 000	145 000	5.0	130 000	131 000	1.0	125 000	130 000	5.0
	Tensile strength, psi	149 000	155 000	6.0	139 000	144 000	5.0	135 000	137 000	2.0
	Elongation, per cent	16.4	14.1	−2.3	15.7	13.2	−2.5	15.0	12.9	−2.1
	Reduction of area, per cent	44.1	42.9	−1.2	38.0	33.6	−4.4	32.6	26.8	−5.8
	Impact, ft-lb	20.3	12.7	−7.6	24.9	14.0	−10.9	20.1	13.5	−6.6
6	0.1 yield strength, psi	137 000	143 000	6.0	129 000	132 000	3.0	125 000	129 000	4.0
	Tensile strength, psi	145 000	152 000	7.0	138 000	143 000	5.0	132 000	135 000	3.0
	Elongation, per cent	15.4	13.7	−1.7	15.0	11.7	−3.3	15.4	11.7	−3.7
	Reduction of area, per cent	42.6	43.2	0.6	37.8	28.4	−9.4	32.8	27.9	−4.9
	Impact, ft-lb	25.1	11.4	−13.7	21.1	13.6	−7.5	19.8	14.7	−5.1

[a] Values are the average of two specimens.

[b] Code:

U = As received (mill annealed).

1, *2*, and *3* = Solution treatment 1550 F for 1 hr, water quenched; age time 16 hr, air cooled.

4, *5*, and *6* = Solution treatment 1650 F for 1 hr, water quenched; age time 1 hr per in. diameter, air cooled

1 and *4* = Age temperature 950 F.

2 and *5* = Age temperature 1050 F.

3 and *6* = Age temperature 1150 F.

TABLE IV.—LONGITUDINAL AND TRANSVERSE CORE MECHANICAL PROPERTIES FOR Ti-6.5Al-3Mo-1V ALLOY.[a]

Condition[b]	Property	2-in. Section			3-in. Section			4-in. Section			6-in. Section		
		Longitudinal	Transverse	Δ	Longitudinal	Transverse	Δ	Longitudinal	Transverse	Δ	Longitudinal	Transverse	Δ
X	0.1 yield strength, psi	126 000	119 000	−7.0	126 000	124 000	−2.0	116 000	116 000	...	110 000	121 000	11.0
	Tensile strength, psi	146 000	146 000	...	144 000	145 000	1.0	138 000	138 000	...	134 000	140 000	6.0
	Elongation, per cent	15.4	19.6	4.2	13.6	13.3	−0.3	14.0	13.3	−0.7	13.3	11.5	−1.8
	Reduction of area, per cent	26.6	34.7	8.1	26.1	26.2	0.1	27.5	28.0	0.5	22.0	17.1	−4.9
	Impact, ft-lb	22.0	13.2	−8.8	25.0	16.7	−8.3	22.2	15.2	−7.0	22.0	15.3	−6.7
A	0.1 yield strength, psi	142 000	145 000	3.0	131 000	136 000	5.0	134 000	138 000	4.0	127 000	138 000	11.0
	Tensile strength, psi	156 000	165 000	9.0	145 000	154 000	9.0	149 000	155 000	6.0	143 000	151 000	8.0
	Elongation, per cent	12.9	7.5	−5.4	12.5	7.1	−5.4	11.1	10.8	−0.3	12.6	10.0	−2.6
	Reduction of area, per cent	18.3	16.6	−1.7	16.1	18.0	1.9	17.1	18.9	1.8	17.6	16.8	−0.8
	Impact, ft-lb	11.8	8.8	−3.0	11.7	9.3	−2.4	13.0	9.6	−3.4	11.8	9.2	−2.6
B	0.1 yield strength, psi	137 000	140 000	3.0	129 000	133 000	4.0	131 000	138 000	7.0	129 000	137 000	8.0
	Tensile strength, psi	147 000	153 000	6.0	138 000	147 000	9.0	144 000	149 000	5.0	139 000	147 000	8.0
	Elongation, per cent	14.3	11.7	−2.6	12.2	11.7	−0.5	15.3	12.9	−2.4	14.7	11.8	−2.9
	Reduction of area, per cent	26.0	23.0	−3.0	16.9	23.2	6.3	25.5	23.1	−2.4	21.1	21.1	...
	Impact, ft-lb	13.8	10.4	−3.4	13.9	11.5	−2.4	15.2	11.1	−4.1	16.0	9.7	−6.3
C	0.1 yield strength, psi	130 000	136 000	6.0	127 000	129 000	2.0	127 000	133 000	6.0	124 000	131 000	7.0
	Tensile strength, psi	139 000	148 000	9.0	134 000	142 000	8.0	137 000	142 000	5.0	133 000	141 000	8.0
	Elongation, per cent	14.0	13.3	−0.7	12.8	11.7	−1.1	16.1	14.3	−1.8	16.1	12.1	−4.0
	Reduction of area, per cent	27.2	24.9	−2.3	17.5	26.9	9.4	27.4	28.7	1.3	24.3	21.6	−2.7
	Impact, ft-lb	14.1	10.6	−3.5	14.2	12.1	−2.1	16.2	12.9	−3.3	15.9	11.5	−4.4
D	0.1 yield strength, psi	149 000	137 000	−12.0	139 000	132 000	−7.0	138 000	130 000	−8.0	129 000	130 000	1.0
	Tensile strength, psi	174 000	171 000	−3.0	161 000	163 000	2.0	157 000	154 000	−3.0	152 000	152 000	...
	Elongation, per cent	4.3	7.9	3.6	8.6	6.3	−2.3	11.8	9.0	−2.8	11.8	12.1	0.3
	Reduction of area, per cent	10.1	14.5	4.4	13.5	14.5	1.0	24.3	19.1	−5.2	20.6	20.1	−0.5
	Impact, ft-b	11.1	8.4	−2.7	14.5	12.0	−2.5	15.7	12.5	−3.2	12.9	12.1	−0.8
E	0.1 yield strength, psi	152 000	149 000	3.0	141 000	142 000	1.0	137 000	134 000	−3.0	131 000	134 000	3.0
	Tensile strength, psi	172 000	174 000	2.0	156 000	166 000	10.0	153 000	150 000	−3.0	146 000	149 000	3.0
	Elongation per cent	7.9	6.0	−1.9	9.7	7.8	−1.9	9.0	7.9	−1.1	11.0	9.3	−1.7
	Reduction of area, per cent	12.2	10.2	−2.0	15.3	15.7	0.4	13.8	11.1	−2.7	19.4	15.8	−3.6
	Impact, ft-lb	12.1	8.7	−3.4	14.7	11.5	−3.2	15.1	12.0	−3.1	17.0	13.0	−4.0
F	0.1 yield strength, psi	149 000	144 000	−5.0	141 000	138 000	−3.0	134 000	136 000	2.0	123 000	136 000	13.0
	Tensile strength, psi	167 000	166 000	−1.0	158 000	152 000	−6.0	149 000	150 000	1.0	139 000	144 000	5.0
	Elongation, per cent	10.0	7.9	−2.1	10.7	10.2	−0.5	10.7	10.4	−0.3	10.7	10.4	−0.3
	Reduction of area, per cent	16.8	13.4	−3.4	16.0	18.0	2.0	18.5	18.1	−0.4	18.6	17.6	−1.0
	Impact, ft-lb	13.0	10.2	−2.8	15.5	11.4	−4.1	15.9	11.2	−4.7	17.2	12.0	−5.2

[a] Values are the average of two specimens.

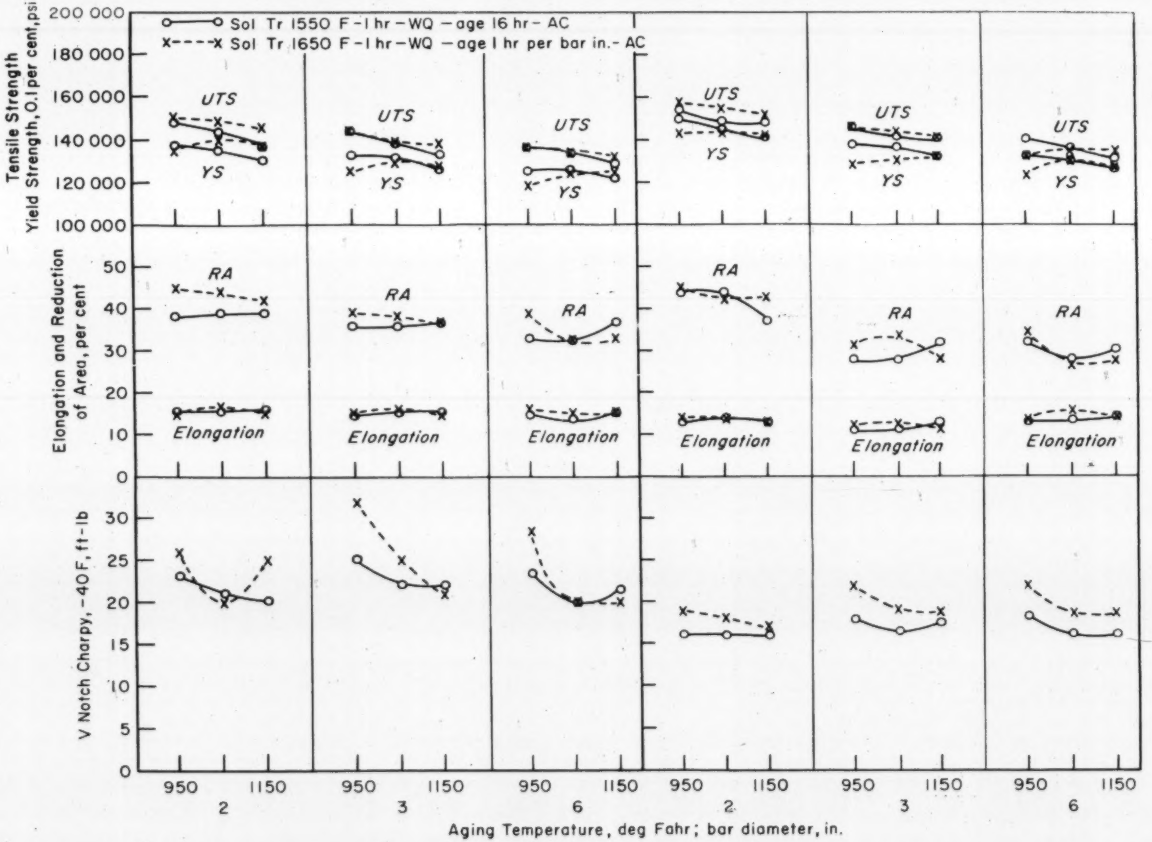
[b] Code:
X = As received (mill annealed).
A, *B*, and *C* = Solution treatments 1650 F for 1 hr, water quenched; age time 24 hr, air cooled.
D, *E*, and *F* = Solution treatments 1760 F for 1 hr, water quenched; age time 1 hr per in. diameter, air cooled.
A and *D* = Age temperature 1000 F.
B and *E* = Age temperature 1100 F.
C and *F* = Age temperature 1200 F.

TABLE V.—LONGITUDINAL AND TRANSVERSE CORE MECHANICAL PROPERTIES FOR Ti-6Mo-3Al ALLOY.[a]

Condition[b]	Property	2-in. Section			3-in. Section			4-in. Section			6-in. Section		
		Longitudinal	Transverse	Δ	Longitudinal	Transverse	Δ	Longitudinal	Transverse	Δ	Longitudinal	Transverse	Δ
Y	0.1 yield strength, psi	116 000	109 000	−7.0	108 000	108 000	...	106 000	111 000	5.0	101 000	105 000	4.0
	Tensile strength, psi	138 000	138 000	...	132 000	134 000	2.0	128 000	127 000	−1.0	128 000	129 000	1.0
	Elongation, per cent	16.8	13.3	−3.5	19.0	15.7	−3.3	17.1	15.0	−2.1	15.3	14.3	−1.0
	Reduction of area, per cent	35.8	31.5	−4.3	47.8	36.7	−11.1	44.4	38.0	−6.4	29.6	28.2	−1.4
	Impact, ft-lb	24.4	18.3	−6.1	27.8	17.7	−10.1	30.2	20.5	−9.7	26.5	18.6	−7.9
G	0.1 yield strength, psi	138 000	152 000	14.0	126 000	135 000	9.0	119 000	127 000	−8.0	115 000	122 000	7.0
	Tensile strength, psi	194 000	195 000	1.0	176 000	171 000	−5.0	164 000	159 000	−5.0	149 000	154 000	5.0
	Elongation, per cent	5.0	4.2	−0.8	7.8	3.1	−4.7	8.6	2.9	−5.7	6.4	5.0	−1.4
	Reduction of area, per cent	6.9	6.1	−0.8	11.1	5.4	−5.7	12.2	4.9	−7.3	11.7	9.8	−1.9
	Impact, ft-lb	4.5	3.9	−0.6	4.9	4.5	−0.4	7.3	5.7	−1.6	9.9	8.1	−1.8
H	0.1 yield strength, psi	136 000	134 000	−2.0	124 000	127 000	3.0	118 000	121 000	3.0	112 000	119 000	7.0
	Tensile strength, psi	187 000	183 000	−4.0	168 000	170 000	2.0	153 000	156 000	3.0	144 000	149 000	5.0
	Elongation, per cent	3.9	3.9	...	7.1	6.3	−0.8	7.9	2.1	−5.8	8.3	6.0	−2.3
	Reduction of area, per cent	10.9	5.4	−5.5	12.0	6.9	−5.1	10.9	4.9	−6.0	13.0	10.3	−2.7
	Impact, ft-lb	5.0	4.2	−0.8	6.0	5.3	−0.7	6.9	3.4	−3.5	11.7	8.5	−3.2
J	0.1 yield strength, psi	131 000	127 000	−4.0	118 000	120 000	2.0	115 000	115 000	...	111 000	118 000	7.0
	Tensile strength, psi	168 000	161 000	−7.0	149 000	147 000	−2.0	140 000	141 000	1.0	138 000	140 000	2.0
	Elongation, per cent	10.4	9.0	−1.4	10.0	6.3	−3.7	13.3	4.3	−9.0	11.8	5.4	−6.4
	Reduction of area, per cent	21.9	16.9	−5.0	15.6	16.7	1.1	18.1	11.9	−6.2	17.6	11.1	−6.5
	Impact, ft-lb	7.4	6.1	−1.3	9.5	7.6	−1.9	12.1	10.4	−1.7	15.2	11.8	−3.4
K	0.1 yield strength, psi	152 000	152 000	...	133 000	140 000	7.0	127 000	131 000	4.0	113 000	116 000	3.0
	Tensile strength, psi	203 000	...	...	177 000	179 000	2.0	161 000	157 000	−4.0	149 000	152 000	3.0
	Elongation, per cent	4.7	...	...	7.1	6.0	−1.1	8.6	2.9	−5.7	7.5	5.0	−2.5
	Reduction of area, per cent	7.1	...	...	11.1	6.9	−4.2	12.4	3.3	−9.1	10.6	9.6	−1.0
	Impact, ft-lb	4.0	3.2	−0.8	7.2	5.8	−1.4	7.1	5.6	−1.5	11.5	8.6	−2.9
L	0.1 yield strength, psi	155 000	159 000	4.0	138 000	138 000	...	125 000	129 000	4.0	112 000	114 000	2.0
	Tensile strength, psi	184 000	193 000	9.0	171 000	171 000	...	164 000	158 000	−6.0	144 000	149 000	5.0
	Elongation, per cent	1.8	2.3	0.5	5.4	4.7	−0.7	10.0	3.2	−6.8	6.8	5.7	−1.1
	Reduction of area, per cent	2.7	3.2	0.5	8.5	6.9	−1.6	16.1	4.9	−11.2	10.9	9.2	−1.7
	Impact, ft-lb	5.0	4.2	−0.8	7.0	5.5	−1.5	8.4	7.4	−1.0	11.5	9.0	−2.5
M	0.1 yield strength, psi	159 000	157 000	−2.0	137 000	134 000	−3.0	125 000	127 000	2.0	112 000	117 000	5.0
	Tensile strength, psi	190 000	193 000	3.0	165 000	165 000	...	154 000	158 000	4.0	139 000	142 000	3.0
	Elongation, per cent	4.7	4.3	−0.4	8.6	4.7	−3.9	8.9	5.0	−3.9	5.7	5.0	−0.7
	Reduction of area, per cent	8.2	6.4	−1.8	15.0	7.2	−7.8	15.2	8.2	−7.0	9.2	9.0	−0.2
	Impact, ft-lb	6.0	5.3	−0.7	10.3	7.4	−2.9	11.7	8.5	−3.2	12.1	10.6	−1.5

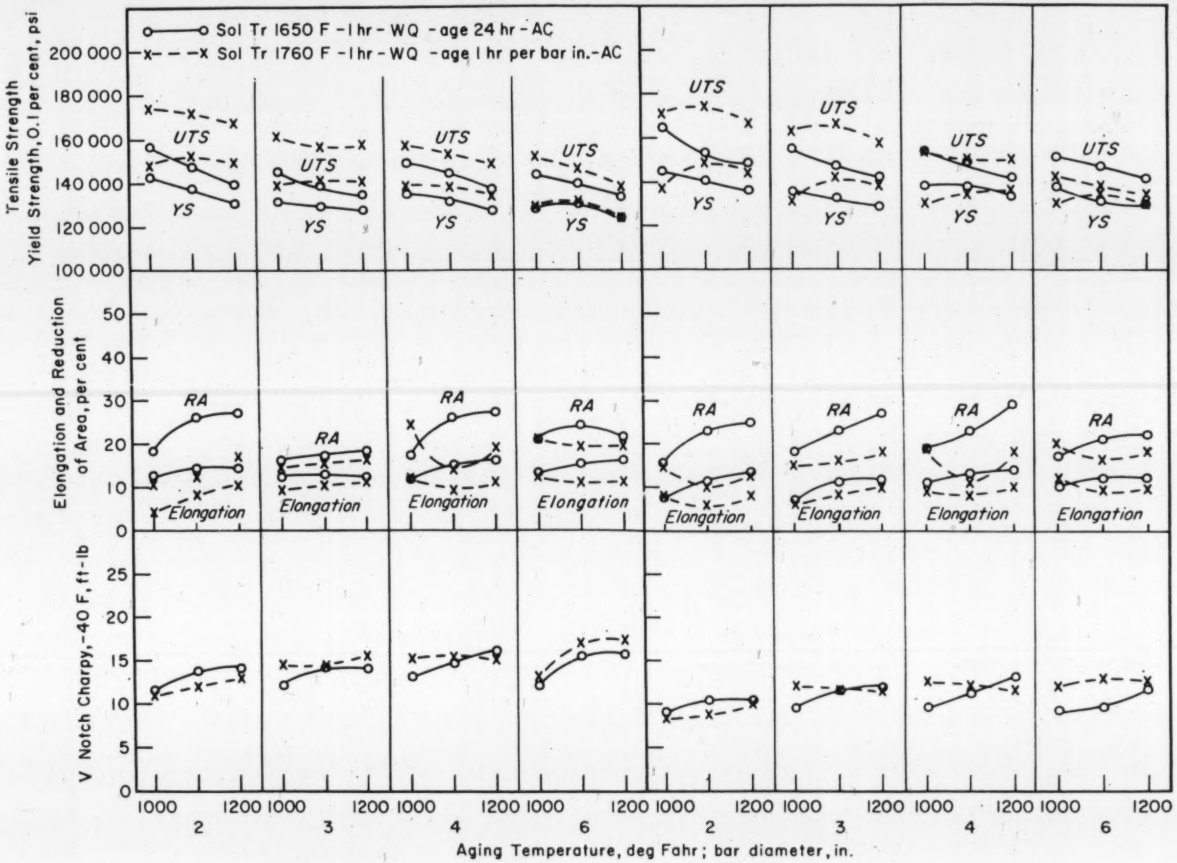
[a] Values are average of two specimens.

[b] Code:
Y = As received (mill annealed).
G, *H*, and *J* = Solution treatment 1550 F for 1 hr, water quenched; age time 24 hr, air cooled.
K, *L*, and *M* = Solution treatment 1625 F for 1 hr, water quenched; age time 1 hr per in. diameter, air cooled.
G and *K* = Age temperature 800 F.
H and *L* = Age temperature 900 F.
J and *M* = Age temperature 1000 F.



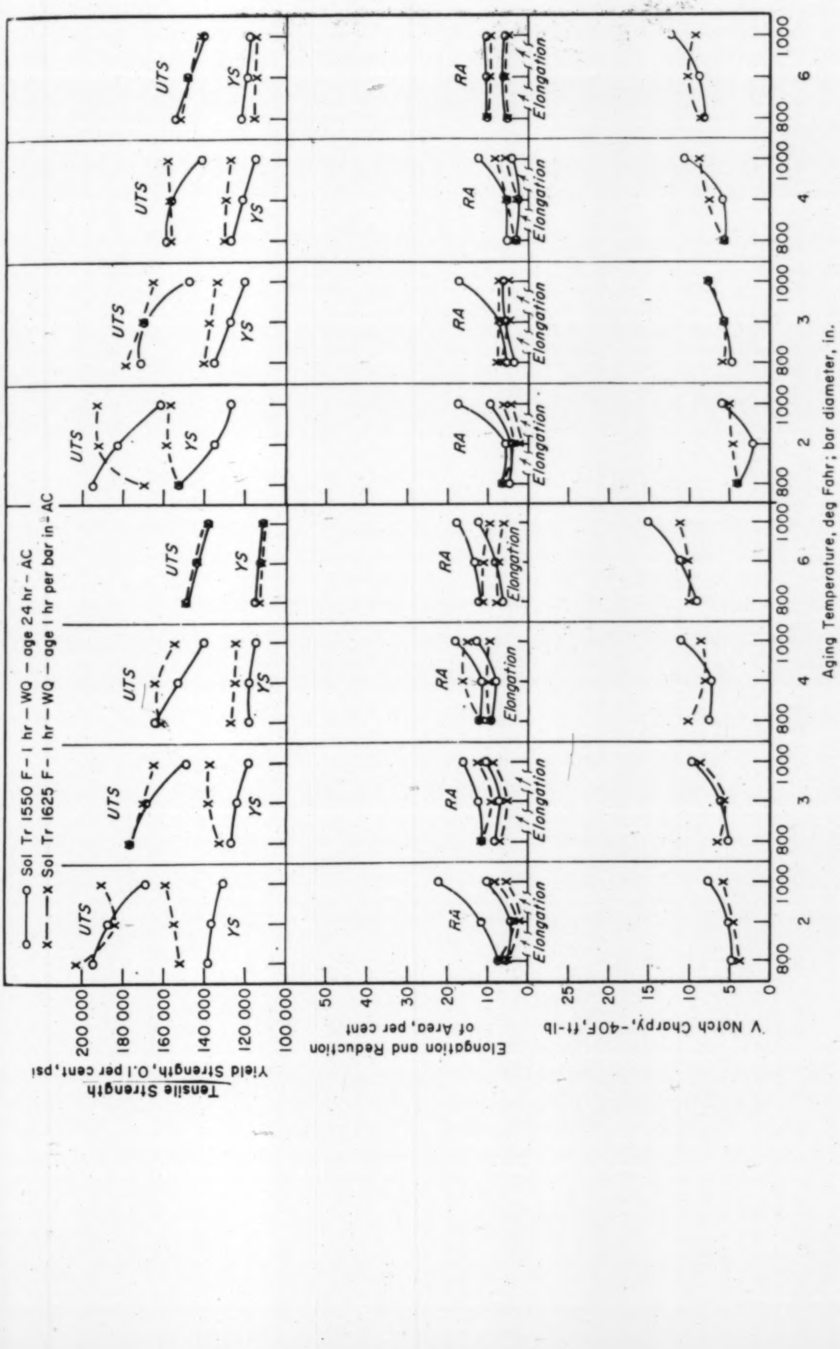
(a) Longitudinal core. (b) Transverse core.

FIG. 1.—Mechanical Properties of the Ti-6Al-4V Titanium Alloy (see Table III).



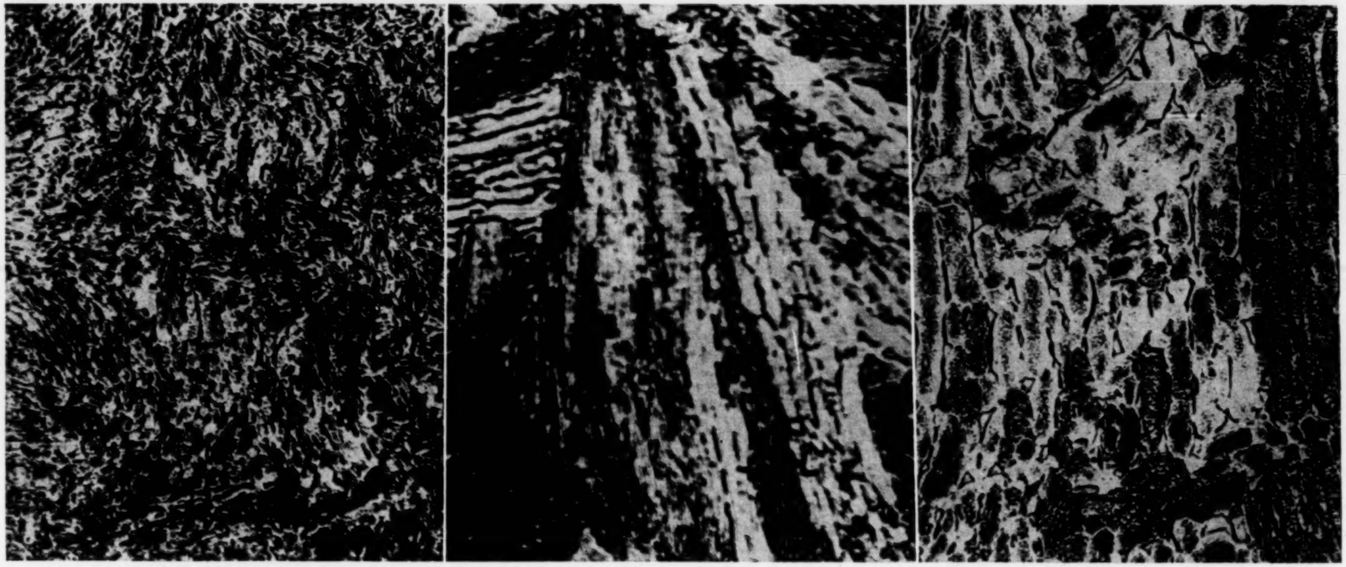
(*a*) Longitudinal core. (*b*) Transverse core.

FIG. 2.—Mechanical Properties of the Ti-6.5Al-3Mo-1V Titanium Alloy (see Table IV).



(a) Longitudinal core. (b) Transverse core.

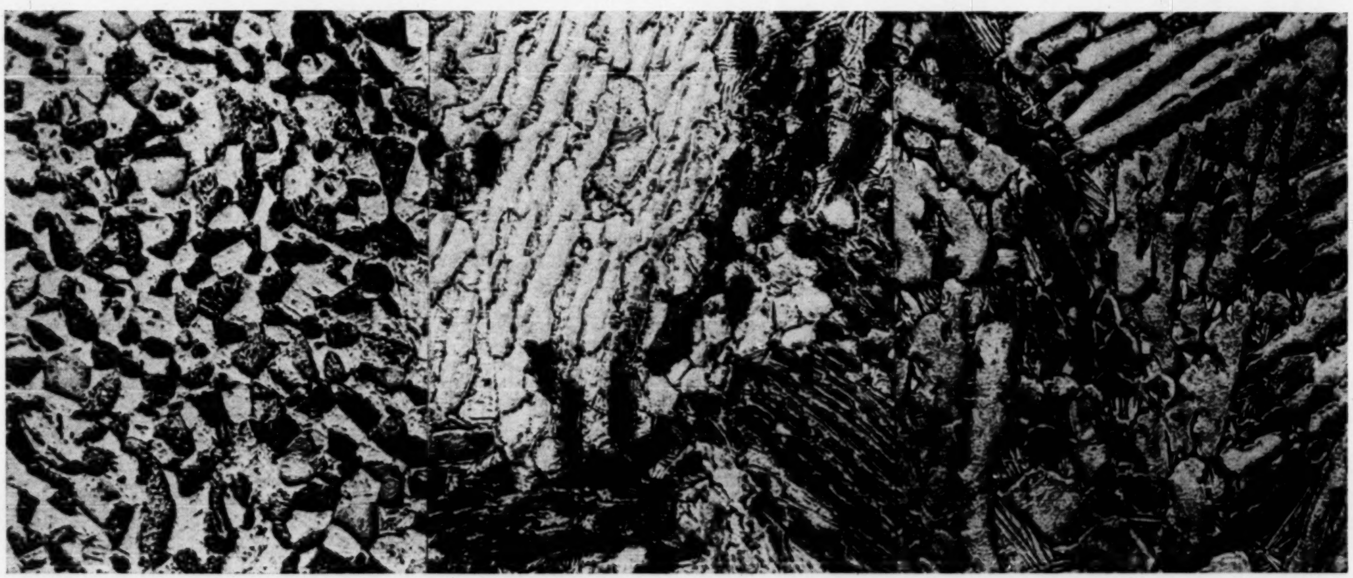
FIG. 3.—Mechanical Properties of the Ti-6Mo-3Al Titanium Alloy (see Table V).



(*a*) 2-in. diameter section. (*b*) 3-in. diameter section. (*c*) 6-in. diameter section.

FIG. 4.—Ti-6Al-4V Alloy As-Received; Annealed Condition (×1000).

Microstructures are predominantly alpha with varying amounts of retained or transformed beta. Etchant: 20 ml benzalkonium chloride, 40 ml glycerin, 38 ml alcohol, and 10 ml hydrofluoric acid.



(*a*) 2-in. diameter section. (*b*) 3-in. diameter section. (*c*) 6-in. diameter section.

FIG. 5.—Ti-6Al-4V Alloy After Treatment: 1650 F Solution Treatment, Aged at 950 F for 1 hr per in., Air Cooled (×1000).

Microstructures are predominantly alpha with varying amounts of retained or transformed beta. Etchant: 20 ml benzalkonium chloride, 40 ml glycerin, 38 ml alcohol, and 10 ml hydrofluoric acid.

properties of the Ti-6Al-4V alloy (Table III) were increased the least amount when heat treated, while the Ti-6.5Al-3Mo-1V (Table IV) and Ti-6Mo-3Al alloys (Table V) were affected to a much larger degree. For example, the maximum increase of ultimate tensile strength in the 2-in. section for the Ti-6Al-4V alloy was 12,000 psi, while the latter two alloys showed increases of 28,000 and 65,000 psi respectively. It is important to note, however, that the increase in strength for the Ti-6Al-V4 alloy was not obtained at the expense of ductility and impact, whereas in the Ti-6.5Al-3Mo-1V and Ti-6Mo-3Al alloys there was a decrease in these properties.

As for the effect of varying the heat treatment temperature for a given alloy, the Ti-6Al-4V alloy (Figs. 1(*a*) and (*b*) was found to be the least sensitive, with the maximum tensile strength difference developed by any two heat treatments being in the order of only 9000 psi. The Ti-6.5Al-3Mo-1V (Figs. 2(*a*) and (*b*)) and Ti-6Mo-3Al alloys (Figs. 3(*a*) and (*b*)) were much more sensitive to heat treatments, and a strength differential of 35,000 psi was developed for both alloys by varying the heat treatment.

Each of the above conditions has its advantage, but also its limitations. Heat treatment temperatures are not critical for the Ti-6Al-4V alloy; however, it is impossible to develop a wide range of properties within the alloy. As for the latter two alloys, a wide property range is possible from heat treating, but if a specific level is desired the heat treatment variables become very critical.

Using a strength-impact criterion, it appears that the higher solution treatment with accompanying aging results in highest properties for each of the alloys. Optimum aging temperatures for Ti-6Al-4V, 6.5Al-3Mo-1V, and 6-Mo-3Al alloys were 950, 1100, and 900 F respectively.

It was found that the general morphology of the microstructure was unchanged by heat treatment except for a slight change in α particle size. This is probably true for most α-β titanium alloys. Figures 4(*a*) to (*c*) are microstructures for the Ti-6Al-4V alloy in the as-received annealed condition, while Figs. 5(*a*) to (*c*) show typical structures in the heat treated condition.

Effect of Section Size:

Section size appears to be the most important of the investigated variables. Ti-6Al-4V again was the least sensitive of the three alloys, with the strength variation between the 2- and 6-in. sections being in the order of 10,000 to 20,000 psi. This is in agreement with similar work published by Sherman and Keller (1) and Titanium Metals Corp. of America (2). The variation for the Ti-6.5Al-3Mo-1V alloy is dependent upon the heat treatment. The lower solution treatment and long aging resulted in a difference about equal to that of Ti-6Al-4V; however, the high solution treatment and accompanying short aging gave a difference of nearly 30,000 psi between the 2- and 6-in. sections. The Ti-6Mo-3Al alloy exhibited the greatest sensitivity to section size—over 50,000 psi in some cases.

It appears that there are two primary factors—microstructure and hardenability—which must be considered in a discussion of section size. Since the sections of smaller diameter received more work during forging, a qualitative change in microstructure is expected, as shown in Figs. 4(*a*) to (*c*). This finer microstructure would be reflected in higher strength, as was observed. Furthermore, as will be discussed in the next section, a comparison of core and midwall strength properties showed the latter to be relatively greater. The magnitude of this difference did not vary in any consistent manner with bar size, and was less than the difference between bars of different sizes. Therefore it is felt that for the size range studied, the microstructure played an important role in determining properties.

On the other hand, the mechanical properties suggested a smoother variation of properties with section size than the microstructure showed, and the unreported midwall strengths were slightly higher than the core values. Thus the presence of a hardenability effect must also be recognized. It would have been desirable to separate the effect of microstructure and hardenability by machining the larger size stock to smaller diameters and then heat treating some of the resultant smaller sizes. Unfortunately, a shortage of material precluded this. Wruck (3) has done similar work on Ti-6Al-4V and Ti-155A alloys. He concludes that the decrease in strength with increasing section size is due to the slower cooling rates obtained as section size increases, and also to the prior microstructure of the heat-treated sections.

Directionality and Location:

A variation in properties with direction was found in all the investigated alloys. Such a directionality is fairly typical of titanium alloys and is caused, in part at least, by preferred orientation.

Of the alloys investigated Ti-6Al-4V showed the largest degree of anisotropy. Transverse yield and tensile strengths were higher than longitudinal values by more than 10,000 and 5000 psi (Table III), respectively. In general there was little difference between the directional strength properties of the other two alloys. This is likely due to the increased β content. A large and consistent difference was also found for impact strength for the Ti-6Al-4V alloy where longitudinal values averaged about 9 ft-lb higher than the transverse. The Ti-6.5Al-3Mo-1V and Ti-6Mo-3Al alloys showed this same trend but in a lesser degree. Longitudinal ductility was generally higher for all alloys, but by inconsistent amounts.

From the data obtained in this investigation, it appears that the degree of anisotropy varies with both heat treatment and section size. Since the preferred orientation aspect was not included in the scope of the project, it was not pursued beyond confirming its presence.

TABLE VI.—OPTIMUM HEAT TREATMENT FOR ALLOYS TESTED.

Titanium Alloy	Solution Treatment	Aging Treatment
6Al-4V	1650 F for 1 hr, water quench	950 F for 1 hr per in. of diameter, air cool
6.5Al-3Mo-1V	1760 F for 1 hr, water quench	1100 F for 1 hr per in. of diameter, air cool
6Mo-3Al	1625 F for 1 hr, water quench	900 F for 1 hr per in. of diameter, air cool

In regard to specimen location, the following can be said: Data indicate that strength properties tend to be slightly higher at midwall than at core locations.

It also appears that section size does not affect this differential. Work done by Sherman and Kessler (1) produced results which are in agreement with this aspect.

It is of interest to note a difference in the directional characteristics of titanium as compared with those of steel. As stated earlier, strength and impact are the most direction-sensitive of the mechanical properties in titanium. However, as shown by Wells and Mehl (4) in steels, it is ductility and impact which show the greatest difference, and there is little variation in yield and tensile strength. It is also probably true that specimen location is more critical in steel than in titanium.

In steels the directional properties are attributed to the chemical macrosegregation during solidification, the preferred alignment of nonmetallic inclusions in the working direction during fabrication, and in part to the crystallographic preferred orientation. In contrast, however, titanium, which is consumable arc melted, has relatively little heterogeneity and a minimum of insoluble nonmetallic inclusions. Thus, directionality of properties predominantly result from the preferred orientation.

SUMMARY

This investigation was mainly concerned with the effect of heat treatment, section size, directionality, and specimen location on the mechanical properties of annealed 6Al-4V, 6.5Al-3Mo-1V, and 6Mo-3Al titanium alloy stock ranging in size from 2 to 6 in. in diameter.

Heat treatments were varied by altering the solution and aging temperatures. An optimum strength-ductility heat treatment for each alloy, based upon the investigated variables, is shown in Table VI.

Section size proved to be a very important and sensitive variable. With a decrease in section size strength values increased, impact values decreased and, in general, the ductility decreased. The strength effect can be partially accounted for by the finer microstructure present in the smaller size bars. Beyond this, however, there is a hardenability effect, since maximum properties could not be developed in the larger sections.

Directionality was a prime variable, especially in the Ti-6Al-4V alloy, where a marked effect on strength and impact properties was shown. For example, transverse yield and tensile strengths were superior to the longitudinal strengths by about 5000 psi and 10,000 psi, respectively, while longitudinal impact averaged nearly 9 ft-lb higher than the transverse. This directionality was mainly attributed to the presence of a crystallographic preferred orientation.

Location is perhaps the least important of the variables studied. Only minor differences in properties between core and midwall locations were noted.

Acknowledgment:

The author wishes to express his appreciation to E. B. Kula and T. S. DeSisto for their suggestions in this work. He is also indebted to H. Margolin of New York University for his review of the paper.

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BEHAVIOR OF NI-O-NEL NICKEL-IRON-CHROMIUM ALLOY IN INTERGRANULAR CORROSION EVALUATION TESTS

By H. R. Copson,[1] B. E. Hopkinson,[1] and F. S. Lang[1]

Synopsis

Data obtained in standard evaluation tests for intergranular corrosion are presented for several experimental alloy compositions which were made during the development of Ni-o-nel nickel-iron-chromium alloy. In general the results show that intergranular corrosion can be prevented by controlling carbon content, titanium content, grain size, and heat treatment. Nevertheless correlation between results in the Huey test and in a boiling sulfuric acid - ferric sulfate test is poor. Examination by X-ray diffraction and electron microscope techniques indicates that this may be due to the presence of a sigma phase, which increased corrosion in the Huey test. The results of the evaluation tests made it desirable to obtain corrosion test data in a number of acid environments. Specimens from a suitable heat were tested in several acids after a mill anneal, after heating for 1 hr at 1200 F, and after welding. The sensitizing heat treatment had no significant effect on the corrosion in these tests, and there was no increased attack at heat-affected zones near welds. It is concluded that the Huey test is not completely reliable for predicting the corrosion behavior of Ni-o-nel alloy in environments other than boiling 65 per cent nitric acid.

Ni-o-nel nickel-iron-chromium alloy is a relatively new material which contains nominally 38 to 46 per cent nickel, 19.5 to 23.5 per cent chromium, 2.5 to 3.5 per cent molybdenum, 1.5 to 3.0 per cent copper, 1.0 per cent max manganese, 0.50 per cent max silicon, 0.05 per cent max carbon, 0.6 to 1.2 per cent titanium, and balance iron. The alloy was developed for use in a wide variety of corrosive solutions, particularly those containing sulfuric and phosphoric acids. It is stabilized to resist intergranular corrosion by controlling the carbon content, titanium content, grain size, and heat treatment.

The present paper reviews some of the early work on the development of this alloy and discusses the effect of composition and heat treatment on the corrosion behavior in standard evaluation tests for intergranular corrosion resistance. The possibility of a sigma phase contributing to intergranular corrosion in the Huey test has been investigated using X-ray diffraction and electron microscope techniques. Corrosion test data in a number of acid environments are reported, and the corrosion behavior in these tests is compared with that in the standard Huey test. The results are significant in relation to similar work on other alloys (**1**–**4**).[2]

Alloy Composition and Heat Treatments

The compositions of all the alloys discussed in this paper are given in Table I.

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[1] Research Laboratory, The International Nickel Co., Inc., Bayonne, N. J.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

The heat treatments are given in the text and in subsequent tables. All heat treatments were carried out in air. Specimens 2 by 1 in. in size were machined out after heat treatment. The surfaces were ground to a No. 120 grit finish.

Alloys No. 11 and 12 correspond to current mill production. The as-received condition of these two alloys was cold-rolled, mill-annealed, and pickled. Alloy No. 12 was used for the corrosion tests in acid environments. This alloy was used in these tests in three conditions of heat treatment: mill-annealed, heated 1 hr at 1200 F, and welded. The welding was accomplished by placing cross welds on one face by metal-arc welding using $\frac{3}{32}$-in. Ni-o-nel welding electrode 135. The slag was removed by sand blasting and scrubbing with pumice.

TABLE I.—COMPOSITION OF MATERIALS.

Alloy	Composition, per cent								
	Nickel	Chromium	Molybdenum	Copper	Manganese	Silicon	Carbon	Titanium	Iron
No. 1	41.1	19.9	2.92	1.62	0.63	0.30	0.017	0.46	balance
No. 2	41.3	19.8	3.21	1.73	0.57	0.36	0.022	0.48	balance
No. 3	41.1	20.6	3.20	1.78	0.62	0.35	0.023	0.44	balance
No. 4	41.4	20.6	3.30	1.90	0.55	0.39	0.035	0.55	balance
No. 5	43.2	18.6	3.26	1.30	0.55	0.30	0.04	0.47	balance
No. 6	43.3	20.8	3.07	1.70	0.74	0.39	0.05	0.42	balance
No. 7	37.2	20.5	1.98	1.09	0.68	0.28	0.05	0.22	balance
No. 8	41.5	19.6	3.21	1.44	0.96	0.58	0.05	0.63	balance
No. 9	42.6	19.5	3.21	1.52	0.90	0.62	0.04	0.83	balance
No. 10	42.1	19.6	3.23	1.60	0.92	0.59	0.04	1.34	balance
No. 11	42.3	21.6	2.94	1.91	0.69	0.23	0.03	0.69	balance
No. 12	40.0	21.4	3.01	1.70	0.63	0.23	0.03	0.81	balance
No. 13	41.0	19.6	4.03	1.91	0.90	0.40	0.05	0.54	balance
No. 14	40.5	20.8	3.04	2.00	0.51	0.30	0.04	0.47	balance
No. 15	41.1	21.7	3.14	1.86	0.57	0.42	0.03	0.57	balance
No. 16	45.7	17.2	3.11	1.58	0.65	0.28	0.05	0.49	balance

TEST PROCEDURES

All tests in boiling solutions were carried out in 1000-ml Erlenmeyer flasks with a cold finger condenser inserted in the neck of the flask. Either 600 or 750 ml of test solution was used. The specimens were inserted in the boiling solution and taken out of the hot solution after completion of the test period. One specimen was used per flask.

The Huey tests in boiling 65 per cent nitric acid were carried out according to ASTM Recommended Practice A 262.[3] Up to five 48-hr exposure periods were used. Fresh acid was used for each period.

The tests at lower temperatures were carried out according to the procedure in ASTM Method B 185.[4] These tests were at 150 and 86 F. At 150 F, the volume of solution in each glass jar was 3500 ml with two duplicate specimens per 3500 ml. At 86 F the volume was 2000 ml with one specimen per jar. The specimens were moved in a vertical circular path at 16 ft per min. When aeration was used, the air stream was dispersed through the solution by a medium-porosity glass diffusion disk.

All tests on alloy No. 12 were usually

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[3] Recommended Practice for Boiling Nitric Acid Test for Corrosion-Resisting Steels (A 262 – 55 T), 1958 Book of ASTM Standards, Part 3, p. 288.

[4] Method of Total Immersion Corrosion Test of Non-Ferrous Metals (B 185 – 43 T), 1958 Book of ASTM Standards, Part 3, p. 266.

TABLE II.—CORROSION TESTS IN BOILING 65 PER CENT NITRIC ACID.

Group	Alloy	Composition, per cent		Heat Treatment				Corrosion Rate, in. per month				
		Carbon	Titanium	deg Fahr	hr	deg Fahr	hr	1st Period	2nd Period	3rd Period	4th Period	5th Period
I....	No. 1	0.017	0.46	2000	1.5	1200	5	0.0004	0.0004	...	...	...
	No. 2	0.022	0.48	2000	1.5	1200	5	0.0005	0.0006	...	...	...
	No. 3	0.023	0.44	2000	1.5	1200	5	0.0004	0.0005	...	...	...
	No. 4	0.035	0.55	2000	1.5	1200	5	0.0017	0.0098	...	...	...
	No. 5	0.040	0.47	2000	1.5	1200	5	0.0804	0.141	...	...	...
	No. 6	0.044	0.41	2000	1.5	1200	5	0.167	0.227	...	...	...
II....	No. 7	0.05	0.22	2000	1.5	1200	5	0.096	...	...	...	...
	No. 8	0.05	0.63	2000	1.5	1200	5	0.0024	0.0053	0.0055	...	...
	No. 9	0.04	0.83	2000	1.5	1200	5	0.0010	0.0009	0.0010	0.0011	...
	No. 10	0.04	1.34	2000	1.5	1200	5	0.0009	0.0008	0.0009	0.0010	...
III....	No. 4	0.035	0.44	1550	1.5	1200	5	0.0004	0.0007	...	...	...
	No. 4	0.035	0.44	1700	1.5	1200	5	0.0004	0.0006	...	...	...
	No. 4	0.035	0.44	1850	1.5	1200	5	0.0006	0.0025	...	...	...
	No. 4	0.035	0.44	2000	1.5	1200	5	0.0017	0.0098	...	...	...
	No. 4	0.035	0.44	2150	1.5	1200	5	0.0019	0.0104	...	...	...
	No. 4	0.035	0.44	2300	1.5	1200	5	0.0368	0.5	...	...	...
	No. 4	0.035	0.44	2400	1.5	1200	5	0.0248	0.5	...	...	...
IV....	No. 11	0.03	0.69	as-received		...	...	0.0005	0.0004	0.0005	0.0006	0.0008
	No. 11	0.03	0.69	as-received		1200	1	0.0005	0.0004	0.0006	0.0011	0.0013
	No. 11[a]	0.03	0.69	1725	0.3	1200	1	0.0004	0.0003	0.0003	0.0004	...
V....	No. 12	0.03	0.81	as-received		...	...	0.0004	0.0004	0.0005	0.0006	0.0008
	No. 12	0.03	0.81	as-received		1200	1	0.0010	0.0565	0.145	0.124	0.116
	No. 12[a]	0.03	0.81	1725	0.3	1200	1	0.0005	0.0011	0.0029	0.0046	0.0058
	No. 12	0.03	0.81	welded		...	...	0.0011	0.0008	0.0010	0.0011	0.0012
VI....	No. 13	0.05	0.54	1950	1	...	...	0.0008	0.0009	0.0009	0.0008	0.0009
	No. 13	0.05	0.54	cold rolled 40 per cent		...	...	0.0008	0.0008	0.0008	0.0008	0.0008
	No. 13	0.05	0.54	cold rolled 40 per cent		1475	2	0.0006	0.0005	0.0006	0.0008	0.0010
	No. 13	0.05	0.54	cold rolled 40 per cent		1475	24	0.0007	0.0006	0.0007	0.0009	0.0012
	No. 13	0.05	0.54	cold rolled 40 per cent		1475	300	0.0006	0.0008	0.0012	0.0016	0.0017

[a] These specimens of alloys No. 11 and 12 were cold rolled 50 per cent prior to annealing at 1725 F.

made in quadruplicate. Many of the tests on the other alloys involved single specimens.

EFFECT OF COMPOSITION AND HEAT TREATMENT ON STABILIZATION IN THE HUEY TEST

During the development of Ni-o-nel nickel-iron-chromium alloy, a number of experimental compositions were prepared, worked down to cold-rolled strip, and heat treated in different manners. Huey tests were run on this material. Data selected to show the important findings are given in Table II.

The specimens in groups I and II of Table II were annealed at 2000 F for 1.5 hr and then heated at 1200 F for 5 hr. Group I shows the effect of carbon content on corrosion in the Huey test and group II the effect of titanium content. It is clear that excellent behavior can be obtained by balancing the carbon and titanium contents.

TABLE III.—CORROSION RATES IN BOILING SULFURIC ACID - FERRIC SULFATE TEST AND IN HUEY TEST.

Group	Alloy	Heat Treatment				Corrosion Rate, in. per month				
		1		2		Huey Test				Sulfuric Acid-Ferric Sulfate Test
		deg Fahr	hr	deg Fahr	hr	1st Period	2nd Period	3rd Period	4th Period	
I. . . .	No. 14	as-received		. . .	. . .	0.0004	0.0003	0.0004	0.0006	0.0014
	No. 14	1600	1.5	. . .	. . .	0.0004	0.0003	0.0003	0.0002	0.0008
	No. 14	1600	24	. . .	. . .	0.0004	0.0003	0.0003	0.0003	0.0007
	No. 14	1700	1.5	. . .	. . .	0.0004	0.0003	0.0003	0.0004	0.0013
	No. 14	1800	1.5	. . .	. . .	0.0004	0.0003	0.0003	0.0004	0.0012
	No. 14	1800	24	. . .	. . .	0.0004	0.0004	0.0003	0.0004	0.0010
II.	No. 14	as-received		1200	1	0.0004	0.0005	0.0009	0.0021	0.0009
	No. 14	1600	1.5	1200	1	0.0004	0.0006	0.0009	0.0016	0.0015
	No. 14	1600	24	1200	1	0.0004	0.0003	0.0005	0.0010	0.0005
	No. 14	1700	1.5	1200	1	0.0005	0.0005	0.0008	0.0017	0.0009
	No. 16	1800	1.5	1200	1	0.0007	0.0007	0.0012	. . .	0.0036
	No. 14	1800	24	1200	1	0.0003	0.0004	0.0006	0.0013	0.0025
III.	No. 16	1600	1.5	1200	1	0.0007	0.0013	0.0035	0.0051	0.0006
	No. 16	1600	24	1200	1	0.0006	0.0009	0.0025	0.0044	0.0013
	No. 15	1700	1.5	1200	1	0.0004	0.0009	0.0010	0.0058	0.0014
	No. 15	1800	1.5	1200	1	0.0004	0.0005	0.0020	0.0071	0.0031
	No. 6	1800	24	1200	1	0.0006	0.0008	0.0030	0.0079	0.0031
	No. 6	1600	1.5	1200	5	0.0004	0.0007	0.0020	0.0050	0.0007
	No. 14	1600	1.5	1200	5	0.0010	0.0014	0.0034	0.0057	0.0010
	No. 6	1600	24	1200	5	0.0005	0.0007	0.0022	0.0056	0.0009
	No. 14	1600	24	1200	5	0.0004	0.0006	0.0016	0.0039	0.0030
	No. 14	1700	1.5	1200	5	0.0004	0.0005	0.0014	0.0033	0.0006
IV.	No. 15	1700	1.5	1200	5	0.0004	0.0083	0.0346	0.0679	0.0009
	No. 15	1800	1.5	1200	5	0.0012	0.0231	0.0630	0.138	0.0009
	No. 14	1800	24	1200	5	0.0004	0.0179	0.0636	0.0776	0.0021
V.	No. 16	1800	24	1200	1	0.071	. . .	. . .	. . .	0.033
VI.	No. 12	as-received		. . .	. . .	0.0004	0.0004	0.0005	0.0006	0.0007
	No. 12	as-received		1200	1	0.0010	0.0565	0.145	0.124	0.0053
	No. 12	welded		. . .	. . .	0.0011	0.0008	0.0010	0.0011	0.0015

The specimens in group III were annealed for 1.5 hr at a range of temperatures and then heated at 1200 F for 5 hr. The high-temperature annealing treatments were harmful for at least two reasons: (1) they increased the grain size and (2) they dissolved the titanium carbides. The low-temperature heat treatments produced excellent results.

The commercial alloy generally meets the expectations implied by the results in groups I, II, and III. Huey test data for a typical heat, alloy No. 11, are given in group IV. The average corrosion rate of as-received material was 0.0006 in. per month. After a sensitizing heat treatment of 1 hr at 1200 F, the average rate was still only 0.0008 in. per month. The rate was decreased slightly to 0.0004 in. per month by cold rolling and annealing at 1725 F for 20 min prior to the sensitizing heat treatment of 1 hr at 1200 F. Most commercial heats behave similarly.

Huey test data for alloy No. 12 are

given in group V of Table II. The corrosion rates of the as-received material were in the expected range, but after a sensitizing heat treatment of 1 hr at 1200 F the rate of attack was excessive. The corrosion resistance was partly but not completely restored by cold rolling and annealing at 1725 F for 20 min prior to the sensitizing heat treatment. This shows that the as-received material was not in the best condition of heat treatment. The grain size of alloy No. 12 was somewhat coarser than that of alloy No. 11 but the difference was not marked, the actual ASTM grain sizes being 5 to 6 and 7 to 8, respectively.[5] It is of interest that the as-received condition of alloy No. 12 withstood welding operations without showing any accelerated attack in the heat-affected zones during the Huey tests.

BEHAVIOR IN OTHER TESTS FOR SUSCEPTIBILITY TO INTERGRANULAR CORROSION

There are other tests besides the Huey test for evaluating the resistance of stainless steels to intergranular corrosion (3). These may be superior to the Huey test in that they reveal only intergranular corrosion associated with carbides and not that associated with the sigma phase. During the developmental work on Ni-o-nel nickel-iron-chromium alloy, a number of specimens were exposed to the Strauss test, to the nitric-hydrofluoric acid test, and to the boiling sulfuric acid - ferric sulfate test. Only in the latter test, however, were enough tests run for a detailed evaluation. The test was carried out as described in the literature (1) and involved exposing specimens for 120 hr in 600 ml of boiling 50 per cent sulfuric acid in which was dissolved 15 g of hydrated ferric sulfate. Acceptance rates for stainless steels are about twice as high in this test as in the Huey test (1).

Table III gives the results obtained in this test and also Huey test data on the same materials. The results in group I in the table pertain to specimens water quenched from temperatures between 1600 and 1800 F. Corrosion rates in the Huey test were low. Corrosion rates in the boiling sulfuric acid - ferric sulfate test varied between 0.0007 and 0.0014 in. per month.

The results in group II in the table pertain to specimens which had been given a sensitizing heat treatment of 1 hr at 1200 F but which nevertheless had quite low corrosion rates in the Huey test, although the rates were somewhat higher than for the specimens of group I. Corrosion rates in the boiling sulfuric acid - ferric sulfate test varied between 0.0009 and 0.0036 in. per month.

Group III in the table includes specimens which had been given sensitizing heat treatments of 1 and 5 hr at 1200 F and which showed some increase in corrosion rate in the Huey test. Corrosion rates in the boiling sulfuric acid - ferric sulfate test remained in the same range as before, actually between 0.0006 and 0.0031 in. per month.

Group IV includes specimens which had been heated for 5 hr at 1200 F and which showed severe corrosion in the Huey test. Corrosion rates in the boiling sulfuric acid - ferric sulfate test remained between 0.0009 and 0.0021 in. per month.

It might be noted that in groups II, III, and IV in the table all specimens giving rates higher than 0.0015 in. per month in the boiling sulfuric acid - ferric sulfate test had been annealed at 1800 F (with one exception). Similarly all specimens in these groups which had been annealed at 1800 F had rates greater than 0.0020 in. per month (with one exception). The reason for this behavior is not known.

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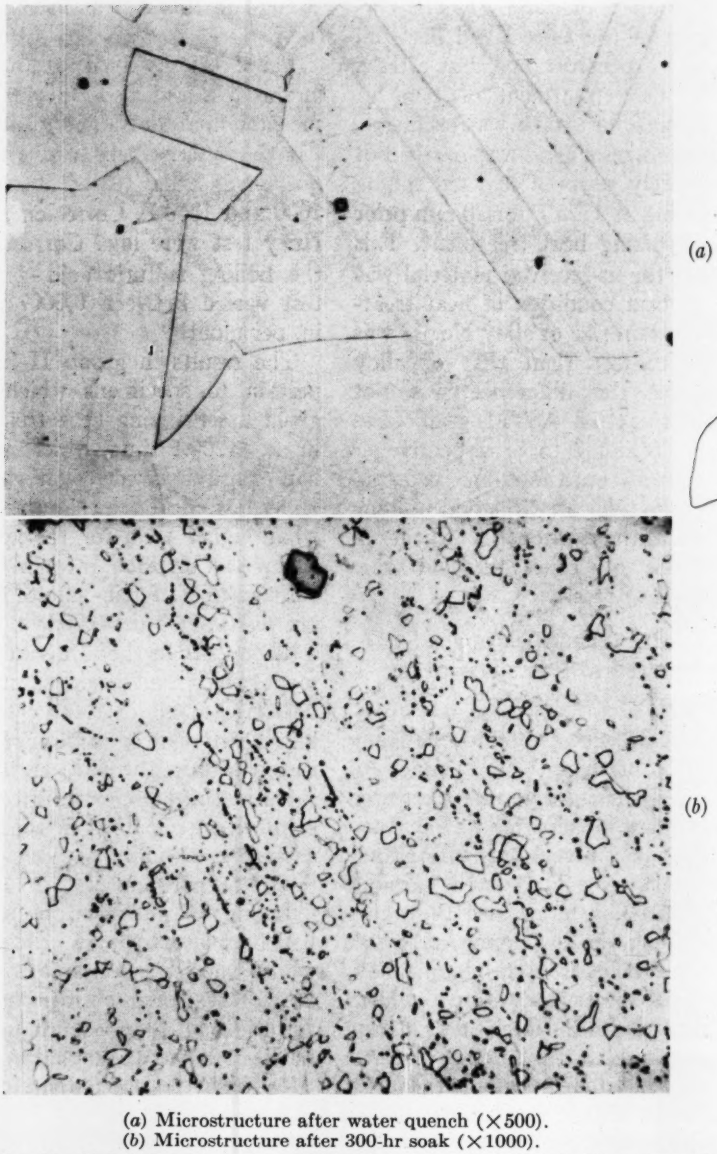
[5] Metals Handbook, 1948 Edition, Am. Soc. Metals, Cleveland, Ohio, pp. 399–405.



(*a*) Microstructure after water quench (×500).
(*b*) Microstructure after 300-hr soak (×1000).

FIG. 1.—Alloy No. 13 Solution Treated at 1950 F for 1 hr, Cold Rolled, Water Quenched, and Soaked 300 hr at 1475 F.

Group V showed extremely severe attack in the Huey test and also in the boiling sulfuric acid - ferric sulfate test.

Group VI in Table III pertains to alloy No. 12. In the as-received condition and after welding this alloy performed well in both tests. After a sensitizing heat treatment of 1 hr at 1200 F, corrosion rates in the Huey test were high as already discussed. In the boiling sulfuric acid - ferric sulfate test this heat treatment increased the rates somewhat above those of groups II, III, and IV but not as high as group V.

It is clear that there is no exact correlation between the two tests. By analogy with the behavior of type 316ELC stainless steel, the results suggest that an intergranular sigma phase may be involved in some of the high rates in the Huey test.

EXAMINATION FOR SIGMA PHASE

An investigation was made on one experimental heat to determine if the sigma phase could be formed in alloys of this general type. Since the Ni-o-nel alloy was being developed for use as a corrosion-resistant material in aqueous solutions, there was little concern about the formation of sigma phase during prolonged exposure at high temperatures, but it was felt that the results might be of some interest. Specimens from alloy No. 13 were water quenched from 1950 F, cold rolled 50 per cent, and then soaked at 1475 F for 2, 24, and 300 hr. Figure 1(*a*) shows the clean structure after the quench, and Fig. 1(*b*) the structure after the 300-hr soak. Here there are distinct large particles of a second phase.

TABLE IV.—X-RAY DIFFRACTION PATTERN ON MATERIAL EXTRACTED FROM ALLOY NO. 13 AFTER HEATING AT 1475 F FOR 300 HR.

X-ray Pattern on Sample		X-ray Pattern on Sigma Phase[a]	
Interplanar Spacing, Å	Intensity[b]	Interplanar Spacing, Å	Intensity[b]
2.30	MW	2.28	VW
2.15	S	2.13	S
2.09	M	2.07	M
2.04	MW	2.02	W
1.991	S	1.969	MW
1.954	S	1.929	S
1.905	M	1.885	M

[a] Powder prepared from high-purity vacuum-melted alloy containing 50 per cent iron and 50 per cent chromium, sigmatized at 1400 F.

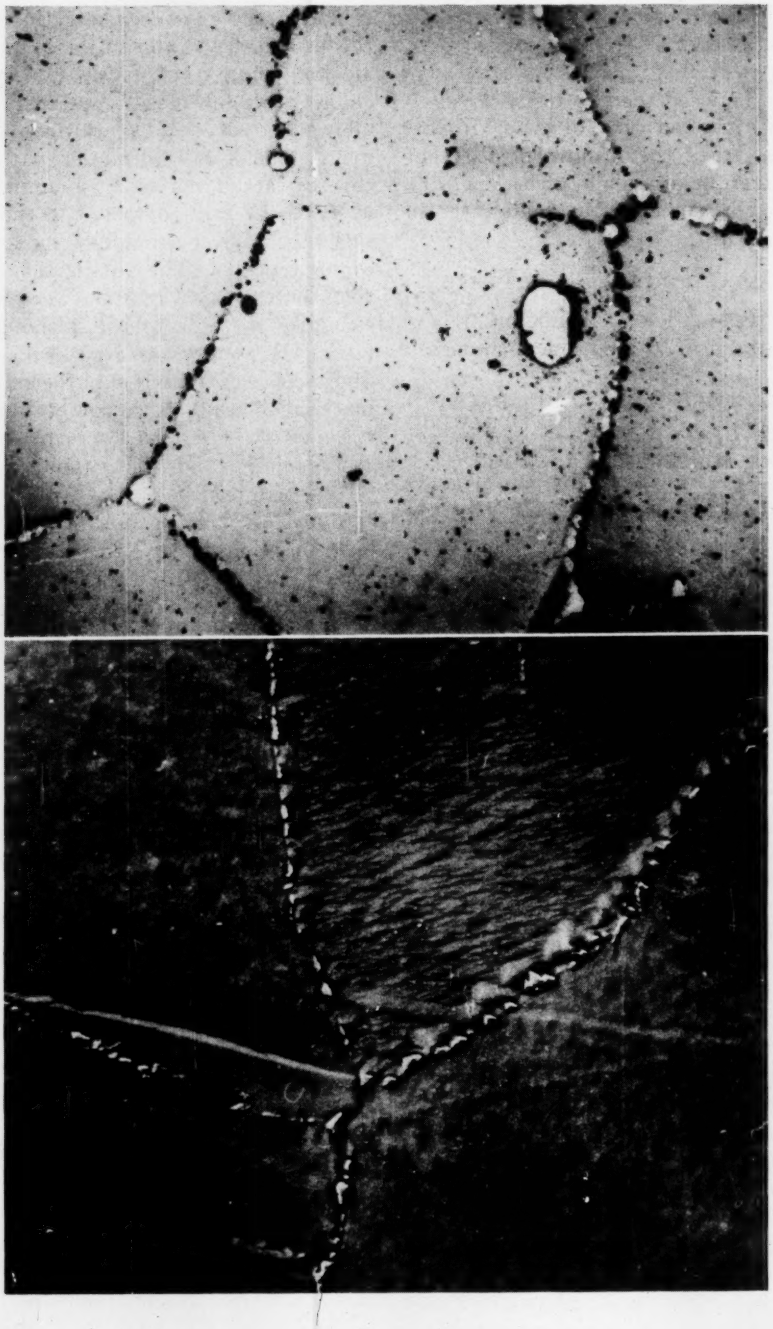
[b] S = Strong, M = Medium, W = Weak.

To extract or concentrate this phase, the specimen was etched for 24 hr in an alcohol solution of picric and hydrochloric acids and the accumulated residue allowed to dry on the surface of the metal. The X-ray diffraction pattern of this extracted material is summarized in Table IV and compared to that of a specimen of iron-chromium sigma phase of high purity. The comparison shows that the extracted material is similar in structure to sigma phase, although the lattice is somewhat expanded. X-ray fluorescence examination of the extracted material showed that in comparison with the matrix, the phase is enriched in molybdenum and chromium and impoverished in nickel.

Huey test data on these specimens are given in group VI of Table II. The presence of large dispersed particles of sigma phase increased the corrosion rate very slightly, but all the rates remained low. Microexamination showed that the corrosion was uniform with no intergranular attack.

Alloy No. 13 is high in molybdenum content which may have helped promote the formation of the sigma phase. Nevertheless the results with this alloy suggest the possibility that intergranular sigma may form under some circumstances and cause attack in the Huey test, similar to the behavior of type 316ELC steel (1–3). Accordingly attempts were made to find sigma in alloy No. 12 after sensitizing heat treatments.

Examinations were made under the electron microscope on as-received material and on specimens heated for 1 and 20 hr at 1200 F. Negative nitrocellulose replicas were stripped from etched specimens and shadow cast with germanium. There was a similar type of precipitate at the grain boundaries in each specimen, but the amount increased with holding time at 1200 F. Figure 2(*a*)



(*a*) Microstructure (×7500).

(*b*) Extraction replica (×5000). Particles of the intergranular phase adhere to the replica.

FIG. 2.—Microstructure of Alloy No. 12 After Heating for 1 hr at 1200 F.

shows the structure of alloy No. 12 after heating for 1 hr at 1200 F.

TABLE V.—CORROSION TESTS ON ALLOY NO. 12 IN SULFURIC ACID.

Test Conditions[a]			Corrosion Rates, in. per yr		
Acid Concentration, per cent	Temperature, deg Fahr	Time, hr	Mill-Annealed	1 hr at 1200 F	Welded
5....	boiling	4	0.035	0.064	0.027
10....	boiling	4	0.056	0.087	0.042
20....	boiling	4	0.197	0.082	0.114
40....	boiling	4	0.135	0.167	0.135
60....	boiling	4	0.531	0.645	0.759
80....	boiling	4	17.0	12.1	11.2
96....	boiling	4	0.829	0.861	0.608
5....	boiling	20	0.028	0.046	0.048
10....	boiling	20	0.051	0.062	0.067
20....	boiling	20	0.034	0.058	0.069
40....	boiling	20	0.065	0.078	0.081
60....	boiling	20	0.324	0.351	0.377
80....	boiling	20	4.0	3.8	4.5
96....	boiling	20	0.213	0.296	0.394
5....	150	4	0.004	0.004	0.006
10....	150	4	0.005	0.010	0.007
20....	150	4	0.009	0.004	0.016
40....	150	4	0.040	0.024	0.051
60....	150	4	0.033	0.025	0.041
80....	150	4	0.053	0.047	0.056
96....	150	4	0.283	0.346	0.264
5....	150	20	0.001	0.001	0.001
10....	150	20	0.001	0.002	0.001
20....	150	20	0.001	0.001	0.001
40....	150	20	0.036	0.038	0.038
60....	150	20	0.030	0.029	0.017
80....	150	20	0.124	0.095	0.021
96....	150	20	0.246	0.249	0.138

[a] Tests at 150 F were aerated and specimens moved at 16 ft per min.

Extraction replicas were obtained from specimens that were deeply etched. Figure 2(*b*) shows the extraction replica obtained from the specimen of Fig. 2(*a*). Particles of the intergranular phase adhered to the replicas. The electron diffraction pattern of this extracted material was obtained and compared with the X-ray pattern for carbides and sigma phase. The results were somewhat inconclusive but there was evidence for the presence of sigma phase and of carbide.

CORROSION BEHAVIOR IN OTHER ACID ENVIRONMENTS

The previous discussion indicates the desirability of running corrosion tests in a number of acid environments to determine whether there is any correlation with behavior in the Huey test. Alloy No. 12 was well suited for this purpose. For convenience these laboratory tests were of shorter duration than might be desirable.

Table V gives the results obtained in sulfuric acid solutions ranging in concentration from 5 to 96 per cent at temperatures of boiling and 150 F and for exposure periods of 4 and 20 hr. The corrosion rates decreased markedly with time due to inhibition by the metal ions accumulating in the solution, and they would be expected to be still lower in longer exposures. This is confirmed by the wide range of usefulness found in plant equipment handling sulfuric acid (**5**).

The sensitizing heat treatment of 1 hr at 1200 F did not appreciably influence the corrosion rate in either the boiling acid or the aerated acid at 150 F. This observation is valid for both the 4- and 20-hr exposures. Comparison with the results on mill-annealed material shows that at the 5 and 10 per cent concentrations in boiling solutions the heat treatment increased the rate somewhat, but at all other concentrations the rates are comparable or lower for heat-treated material. Inspection of the cross-welded specimens showed no increased corrosion in the heat-affected zones.

Table VI gives the results obtained on alloy No. 12 in a number of other acid environments. These comprise boiling phosphoric acid solutions, aerated sulfuric acid solutions at 150 F with additions of nitric acid and sodium chloride, several boiling organic acid solutions, and several acid salt solutions. There was no major difference in the behavior of mill-annealed material and specimens heated for 1 hr at 1200 F. In some tests the corrosion rates were somewhat higher in one condition and in other tests in the other condition. Most of the corrosion rates were quite low. In the three most corrosive test solutions, the corrosion rates were lower after the sensitizing heat treatment. None of the cross-welded specimens showed any signs of increased attack in the heat-affected zones.

TABLE VI.—CORROSION TESTS ON ALLOY NO. 12 IN OTHER ACID ENVIRONMENTS.

Test Conditions			Corrosion Rates, in. per yr		
Environment	Temperature, deg Fahr	Time, hr	Mill-Annealed	1 hr at 1200 F	Welded
Phosphoric acid, 60 per cent	boiling	20	0.008	0.012	0.015
Phosphoric acid, 70 per cent	boiling	20	0.007	0.013	0.018
Phosphoric acid, 80 per cent	boiling	20	0.051	0.038	0.052
10 per cent H_2SO_4[a]	150	20	0.001	0.002	0.001
10 per cent H_2SO_4 + 5 per cent HNO_3[a]	150	20	0.002	0.002	0.002
10 per cent H_2SO_4 + 5 per cent NaCl[a]	150	20	0.203	0.181	0.222
Acetic acid, 10 per cent	boiling	120	<0.001	0.002	<0.001
Formic acid, 10 per cent	boiling	120	0.003	0.003	0.003
Lactic acid, 10 per cent	boiling	120	<0.001	<0.001	<0.001
Maleic acid, 10 per cent	boiling	120	<0.001	<0.001	<0.001
Oxalic acid, 10 per cent	boiling	120	0.020	0.015	0.016
Phthalic acid, 10 per cent	boiling	120	<0.001	<0.001	<0.001
Aluminum chloride,[a] 25 per cent	86	168	0.005	0.005	...
Zinc chloride,[a] 25 per cent	86	168	0.0001	0.0002	...
Sodium bisulfite,[b] 25 per cent	86	168	<0.0001	0.0002	<0.0001

[a] Solutions were aerated with specimens moving at 16 ft per min.

[b] No air was added to solutions; specimens moving at 16 ft per min.

The results in Tables V and VI contrast with those obtained in the Huey test. After 240 hr in boiling 65 per cent nitric acid, the average corrosion rate of alloy No. 12 in the mill-annealed condition was 0.0005 in. per month, and after heating for 1 hr at 1200 F the average rate increased to 0.0939 in. per month, an increase of almost 200 times.

While these laboratory tests were of relatively short duration, the Ni-o-nel alloy has resisted intergranular corrosion for extended exposure times under many service conditions (5). It is concluded therefore that the Huey test is not completely reliable for predicting the effect of sensitizing heat treatments or of welding on the corrosion behavior of Ni-o-nel nickel-iron-chromium alloy in environments other than boiling 65 per cent nitric acid.

Acknowledgments:

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ELEVATED-TEMPERATURE DYNAMIC MODULI OF METALLIC MATERIALS*

By W. H. Hill,[1] K. D. Shimmin,[2] and B. A. Wilcox[2]

Synopsis

Elevated-temperature dynamic moduli of elasticity of 40 commercial metals and alloys at temperatures ranging up to 1800 F are presented. The design, construction, and operation of an electrostatic device for measuring dynamic modulus under conditions of longitudinal resonance are described.

The dynamic modulus measurements were made under conditions of continuous heating at a nominal rate of 12 F per min. The materials tested included aluminum alloys, beryllium, titanium alloys, plain carbon and low-alloy steels, stainless steels, a cobalt alloy, nickel alloys, and refractory metals. Room-temperature comparisons of dynamic moduli with static moduli are given in most instances.

Results of dynamic modulus measurements are presented which show the variations of modulus as a function of temperature and alloy composition in a series of experimental vanadium-titanium alloys.

In the design of load-carrying members subjected to high temperature as well as stress, it is imperative that Young's modulus be known so that deflections can be calculated for any combination of stress and temperature. This property is conventionally determined from the static stress-strain plots of one or more specimens tested at each temperature. This widely accepted method has several disadvantages, the principal one being the large number of carefully prepared specimens needed to establish the temperature-dependence of modulus for a given material.

The investigation reported here employs a less conventional approach to the determination of elastic modulus (Young's modulus) which is based on the relationship between the speed of sound in a material and its elastic modulus. Since the speed of sound in a specimen of a given geometry is manifested in its resonant frequency, it becomes necessary only to determine that property of the material at elevated temperatures to evaluate the elevated-temperature elastic modulus. This determination is obtained nondestructively, thus permitting a large number of determinations at various temperatures with a minimum number of specimens.

The program reported here was conducted for the purpose of evaluating the elevated-temperature dynamic moduli of a wide variety of commercial metals and alloys. In addition, elevated-temperature dynamic moduli were evaluated for a

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series of experimental vanadium-titanium alloys having nominal compositions of 8, 17, 25, 32, and 48 per cent by weight titanium. This system was investigated since it has been shown (**1,2**)[3] that titanium is one of the few additions which do not have a detrimental effect on the ductility of vanadium. In addition to being forgeable, vanadium-titanium alloys show improved elevated-temperature tensile strength over that of pure vanadium. Rostoker (**2**) predicted that high-strength vanadium-base alloys would have to be composed of vanadium-titanium alloys with third and possibly fourth-element additions.

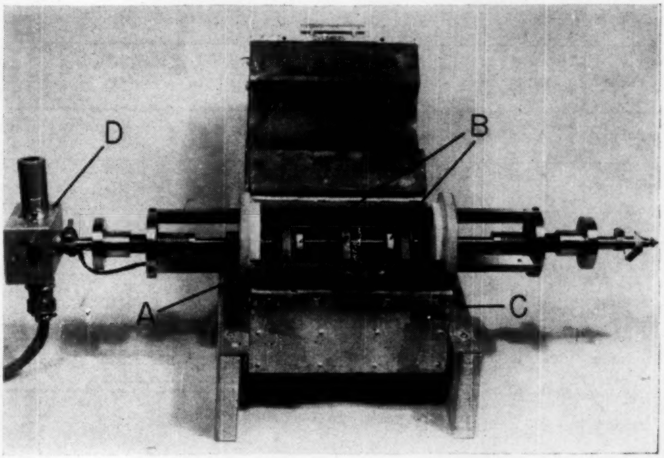

FIG. 1.—Dynamic Modulus Equipment.

A—Specimen, *B*—capacitor plate support assembly, *C*—furnace, and *D*—cathode follower (preamplifier first stage).

EXPERIMENTAL PROCEDURES

Materials:

The commercial materials considered as representative of those receiving general interest for elevated-temperature structural applications were alloys of aluminum, beryllium, copper, titanium, iron, cobalt, and nickel. In addition, several pure metals, including refractory metals, were investigated. Alloys of engineering interest were tested in the condition of heat treatment considered to be of widest application. Detailed informaton regarding the specific heat treatments employed has been reported by Hill and Shimmin (**3**). The compositions of test materials are given in Table I.

The commercial alloys and pure metals, including the calcium-reduced vanadium, were obtained from commercial sources, but the aluminothermic vanadium and the 5 vanadium-titanium alloys were prepared at the Metals and Ceramics Laboratory, Materials Central, Aeronautical Systems Division. These materials were prepared as $\frac{3}{4}$-lb buttons by nonconsumable arc melting, each alloy being melted three or four times. The buttons were sectioned, and the center portions were given approximately 40 per cent reduction by forging at 1850 F. Prior to forging, the specimens were glass coated for protection against oxidation, and an argon atmosphere was

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

TABLE I.—MATERIAL COMPOSITIONS.

Material	Condition	Element, per cent by weight																		
		Aluminum	Carbon	Columbium	Cobalt	Chromium	Copper	Iron	Hydrogen	Manganese	Molybdenum	Nitrogen	Nickel	Oxygen	Phosphorus	Sulfur	Silicon	Titanium	Vanadium	Other
Aluminum and aluminum alloys:																				
High-purity aluminum	recrystallized	99.994	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
2024	T4	balance	...	...	...	0.02	4.30	0.28	...	0.62	...	...	<0.02	...	...	...	0.17	0.02	...	1.32 Mg, 0.06 Zr
7075	T6	balance	...	...	...	0.21	1.34	0.25	...	0.06	...	...	<0.02	...	...	...	0.20	0.06	...	2.44 Mg, 5.39 Zn
Titanium and titanium alloys:																				
Iodide titanium	as-grown	...	0.03	...	...	...	...	0.02	0.009	...	...	0.005	...	0.01	...	...	...	balance	...	...
Ti-75A	annealed	...	0.04	...	...	...	...	0.12	0.032	...	...	0.032	...	0.19	...	...	...	balance	...	...
Ti-6Al-4V	mill annealed	6.0	...	...	...	0.002	...	0.20	...	...	...	...	...	...	...	...	...	balance	3.0	...
B-120 VCA	mill annealed	3.7	...	...	...	10.7	...	...	0.009	...	...	0.02	...	...	...	...	...	balance	12.5	...
Plain carbon and low-alloy steels:																				
SAE 1020	warm rolled	...	0.24	...	...	...	...	balance	...	0.30	...	...	...	...	0.01	0.045	0.01	...	...	...
SAE 4130	normalized and tempered	...	0.24	...	...	0.94	...	balance	...	0.25	0.17	...	...	...	0.01	0.012	0.16	...	...	...
SAE 4340	austenitized and tempered	...	0.41	...	...	0.80	...	balance	...	0.74	0.27	...	1.75	...	0.015	0.015	0.26	...	...	...
LaBelle HT	heat treated^a	...	0.429	...	...	1.31	...	balance	...	1.39	0.38	...	0.13	...	0.013	0.021	0.68	...	0.24	...
Peerless 56	austenitized and tempered	...	0.387	...	...	3.23	...	balance	...	0.47	2.83	...	0.16	...	0.016	0.015	0.70	...	0.24	...
Vascojet 1000	heat treated^a	...	0.41	...	...	5.07	...	balance	...	0.17	1.73	...	...	...	0.019	0.013	0.80	...	0.49	...
Stainless steels:																				
410	tempered	...	0.16	...	...	13.10	...	balance	...	0.45	...	0.03	...	...	0.019	0.019	0.291	...	...	...
AM 350	SCT (850F)	...	0.10	...	...	16.10	...	balance	...	1.15	2.73	...	4.55	...	0.026	0.017	0.216	...	...	...
17-7PH	TH 1050	1.13	0.073	...	...	17.65	...	balance	...	0.61	...	...	7.20	...	0.023	0.012	0.334	...	...	...
PH 15-7 Mo	TH 1050	1.24	0.075	...	...	14.10	...	balance	...	0.70	2.27	...	7.31	...	0.025	0.018	0.390	...	...	...
A-286	SHT, aged	0.30	0.091	...	...	15.47	...	balance	...	0.98	1.14	...	25.93	...	0.021	0.006	0.625	2.30	0.29	...
Nickel and nickel alloys:																				
Electrolytic nickel	as-deposited	...	0.01	...	...	...	...	...	...	<0.004	...	...	balance	...	0.014	0.001	0.01	...	...	...
Inconel	annealed	0.06	0.06	<0.01	...	13.65	...	6.97	...	0.25	...	...	balance	...	...	...	0.226	<0.01	...	...
Inconel W	annealed	0.50	0.028	<0.01	...	15.95	...	6.30	...	0.50	...	...	balance	...	...	...	0.23	2.40	...	...
Inconel X	heat treated^a	1.00	0.06	0.42	...	15.16	...	6.74	...	0.68	...	...	balance	...	...	...	0.30	2.55	...	...
Inconel 700	heat treated^a	3.29	0.135	...	28.98	15.35	...	0.25	...	0.25	3.09	...	balance	...	...	...	0.018	2.28	...	...
Inconel 713-C	as-cast	5.60	...	2.10	...	11.90	...	...	...	...	5.00	...	balance	...	...	...	...	0.52	...	...
M-252	heat treated^a	0.90	0.144	...	9.90	18.95	...	0.75	...	0.02	10.04	...	balance	...	...	...	0.056	2.35	...	...
René 41	heat treated^a	1.63	0.09	...	11.22	18.88	...	<0.30	...	<0.04	9.79	...	balance	...	...	...	0.10	3.21	...	...
Udimet 500	heat treated^a	2.65	0.093	...	18.78	20.16	<0.02	0.64	...	0.02	4.05	...	balance	...	...	...	0.044	3.00	...	...
Udimet 700	wrought, heat treated^a	4.33	0.07	...	18.00	15.50	0.10	0.12	...	0.10	5.00	...	balance	...	...	0.007	0.10	3.50	...	0.032 B, 0.05 Zr

Udimet 700	cast, heat treated[a]	4.70	0.05	...	19.00	14.45	...	0.52	...	<0.10	5.00	...	balance	...	...	0.007	0.02	3.60	...	0.029 B, <0.03 Zr
Nimonic 90	heat treated[a]	1.09	0.033	...	20.00	20.20	...	0.18	...	0.05	<0.01	...	balance	...	...	...	0.39	2.48	...	...
Hastelloy B	heat treated[a]	...	0.046	...	...	0.19	...	6.54	...	0.21	29.10	...	balance	...	...	...	0.15	...	...	...
Waspaloy	heat treated[a]	1.33	0.063	...	13.35	10.28	<0.02	0.66	...	0.02	4.45	...	balance	...	...	...	0.044	2.84	...	...
Miscellaneous alloys:																				
Beryllium copper	SHT, and SHT plus aged	...	...	...	0.13	...	balance	0.12	...	...	...	...	0.008	...	...	...	...	...	...	1.18 Be
S-816	heat treated[a]	...	0.358	2.87	43.40	19.92	...	3.85	...	1.39	3.86	...	19.94	...	...	...	0.38	...	...	4.90 W
Invar	recrystallized	...	...	...	...	...	...	balance	...	...	...	...	35.60	...	...	...	...	...	...	...
Other pure metals:																				
Beryllium	Powder metallurgy	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	98+ Be
Molybdenum	arc melted, recrystallized	...	0.20	...	...	...	...	...	0.0001	...	99+	0.0003	...	0.0014	...	...	...	...	...	...
Tungsten	arc melted, C.W.	...	0.03	...	...	...	...	...	0.0001	...	...	0.0001	...	0.0099	...	...	...	...	...	99+ W
Vanadium	aluminothermic, Rx	...	0.025	...	...	...	...	...	0.0023	...	...	0.0058	...	0.035	...	...	...	...	...	...
Vanadium	calcium-reduced, CW	...	0.05	...	...	...	...	...	0.0032	...	...	0.056	...	0.086	...	...	...	...	...	...
Vanadium-titanium alloys:																				
V-8Ti	hot forged	...	0.04	...	...	...	...	...	0.0022	...	...	0.0028	...	0.033	...	...	...	8.44	balance	...
V-17Ti	hot forged	...	0.04	...	...	...	...	...	0.0051	...	...	0.0043	...	0.044	...	...	...	16.85	balance	...
V-25Ti	hot forged	...	0.04	...	...	...	...	...	0.0064	...	...	0.0036	...	0.050	...	...	...	25.41	balance	...
V-32Ti	hot forged	...	0.03	...	...	...	...	...	0.0065	...	...	0.0034	...	0.047	...	...	...	32.02	balance	...
V-48Ti	hot forged	...	0.03	...	...	...	...	...	0.0073	...	...	0.0032	...	0.037	...	...	...	48.23	balance	...

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[a] Materials were tested in the heat treatment considered to be of widest application. For the exact heat treatments used, refer to reference (3).

used in the heat treating furnace for additional protection. The forgings were machined to cylindrical specimens 3 in. long and $\frac{1}{4}$ in. in diameter. Chemical analyses of these materials are also given in Table I.

Equipment and Techniques:

The determinations of dynamic moduli were made in special equipment designed and assembled at the Metals and Ceramics Laboratory, Materials Central, Aeronautical Systems Division. In the apparatus, shown in Fig. 1, cylindrical specimens 3 in. long were rigidly supported at their midlength, the nodal point for fundamental resonance. The specimens were driven into longitudinal resonance by a pulsating electrostatic force applied at one end. The electrostatic force was obtained by application of about 150 v of the proper drive frequency to a plate parallel to the end of the specimen and adjusted for minimum air gap necessary to prevent discharge. The specimens were at ground potential. The drive voltage originated from a signal generator, was amplified by a 60-w audioamplifier and stepped up by a transformer. At the opposite end of the specimens the same arrangement was used to obtain the response signal. A 300-v bias was applied to the pickup plate and the voltage generated by change in capacitance due to change in gap was amplified 3500 times and fed into an oscilloscope and electronic frequency counter. Since the particular drive transformer being used was not efficient at full resonant frequency, half of the resonant frequency was used for drive purposes. Two electronic frequency counters were used, one counting the drive frequency ($\frac{1}{2}$ the fundamental) and the other counting the pickup frequency (fundamental). The fundamental frequency was established by the maximum amplitude response observed on the oscilloscope. Figure 1 shows the specimen A, capacitor plate support assembly B, furnace C, and cathode follower (preamplifier first stage) D. For temperature indication a thermocouple was tied to the specimen at midlength. A helium atmosphere was introduced for tests on iodide titanium, Ti-75A, vanadium, vanadium-titanium alloys, molybdenum, and tungsten. All other materials were tested in air.

The fundamental resonant frequency, f, determined by this method is related to Young's modulus, E, by the following equation (4):

$$E = \frac{4\rho l^2 f^2 \left(1 + \dfrac{\pi^2\mu^2 r^2}{2l^2}\right)}{\left(1 - \dfrac{1}{8Q^2}\right)^2} \quad \ldots\ldots (1)$$

where:

l = specimen length,
r = specimen radius,
ρ = density,
μ = Poisson's ratio, and
$1/Q$ = expression for internal friction.

Neglecting the Poisson's ratio term and the damping term reduces the equation to

$$E = 4\rho l^2 f^2 \quad \ldots\ldots\ldots\ldots\ldots (2)$$

A slight error is introduced at higher temperatures by neglecting the change in length with temperature. All values of modulus reported herein have been calculated using room-temperature values of length and density. Graft, Levinson, and Rostoker (5) have developed a mathematical derivation of a correction factor to be applied to the modulus formula to obtain elevated-temperature values using room-temperature measurements. The corrected formula appears as follows:

$$E_t = 4\rho l^2 f^2 \left(\frac{1 + 2\alpha T}{1 + 3\alpha T}\right) \quad \ldots\ldots (3)$$

TABLE II.—ROOM-TEMPERATURE PROPERTIES OF COMMERCIAL ALLOYS AND PURE METALS.

Material	Static Modulus, psi	Dynamic Modulus, psi	Rockwell Hardness, B scale	Density, lb per cu in
Aluminum and aluminum alloys:				
High-purity aluminum	...	9.97×10^6	...	0.0975
2024-T4	10.6×10^6	10.78	75	0.1007
7075-T6	10.4	10.65	...	0.1031
Titanium and titanium alloys:				
Iodide titanium	...	12.45	39	0.1630
Ti-75A	...	15.93	28.5[c]	0.1630
Ti-6Al-4V	16.4	16.52	...	0.1596
B-120 VCA	14.0	14.09	...	0.1740
Plain carbon and low-alloy steels:				
SAE 1020	30.2	30.46	61	0.2832
SAE 4130	31.3	30.95	...	0.2830
SAE 4340	29.7	29.68	...	0.2822
LaBelle HT	29.3	29.59	...	0.2770
Peerless 56	30.4	31.28	...	0.2812
Vascojet 1000	30.7	30.70	...	0.2790
Stainless steels:				
410	32.3	32.38	...	0.2788
AM 350	29.6	29.34	...	0.2813
17-7PH	29.2	29.26	...	0.2776
PH 15-7 Mo	29.1	29.39	...	0.2803
A 286	28.8	28.42	...	0.2855
Nickel and nickel alloys:				
Electrolytic nickel	...	29.32	...	0.3220
Inconel	31.6	31.43	69	0.3062
Inconel W	31.0	30.71	80	0.2980
Inconel X	31.6	31.51	...	0.2968
Inconel 700	32.1	32.56	...	0.2918
Inconel 713C	...	30.27	...	0.2856
M-252	30.2	29.07	...	0.2917
René 41	31.9	31.88	...	0.2981
Udimet 500	32.3	32.14	...	0.2901
Udimet 700, W[a]	32.4	32.45	...	0.2866
Udimet 700, C[b]	...	29.67, 30.58	...	0.2865
Nimonic 90	...	33.03	...	0.2949
Hastelloy B	31.4	31.66	...	0.3302
Waspaloy	32.2	32.25	...	0.2963
Miscellaneous alloys:				
Beryllium copper, SHT	17.8	17.94	31	0.3075
Beryllium copper, SHT and aged	17.6	18.02	94	0.3078
S-816	34.9	33.85	23[c]	0.3108
Invar	...	21.70	...	0.2925
Other pure metals:				
Beryllium	...	42.65	...	0.0674
Molybdenum	47.2	46.35	...	0.3690
Tungsten	...	58.56	...	0.6920

[a] W = wrought condition.
[b] C = cast condition.
[c] Rockwell hardness, C scale.

where:

E_t = modulus at temperature t,
α = linear coefficient of thermal expansion,
T = temperature difference, and
l, ρ, and f are as previously defined.

In analysis of the accuracy of dynamic modulus values, errors in measurement should be considered as well as those resulting from the simplifying assumptions used in obtaining Eq 2. An analysis of the errors in modulus determination, excluding the effect of temperature discussed above, indicates that the total error in dynamic modulus values is less than 0.4 per cent.

During dynamic modulus determinations the temperature of the test specimen was measured by a thermocouple tied to the specimen with asbestos string at the midpoint location of the clamping screws. Prior to the test program the temperature distribution along a specimen was established using dummy specimens. These specimens had fine thermocouples welded to the specimen near each end and at the midpoint. Using the dummy specimens, Variac settings for power to the three-zone furnace were established to give the most uniform temperature distribution along the specimens. It is estimated that the maximum spatial temperature deviation was ±4 F and that the accuracy of the indicated temperature was ±3 F.

During the test program simultaneous readings of temperature and resonant frequency were made while the specimens were continuously heated at a nominal rate of 12 F per min. In most instances densities of the test specimens were obtained by using the method of measuring the loss of weight in liquid. In some cases density measurements were made by weighing the specimens and using calculated volume.

The room-temperature static modulus determinations were made using a 20,000-lb Universal testing machine for loading and Tuckerman optical strain gages for strain measurement. A comparison of room-temperature static moduli and dynamic moduli (Table II) indicates that good agreement is realized in most cases.

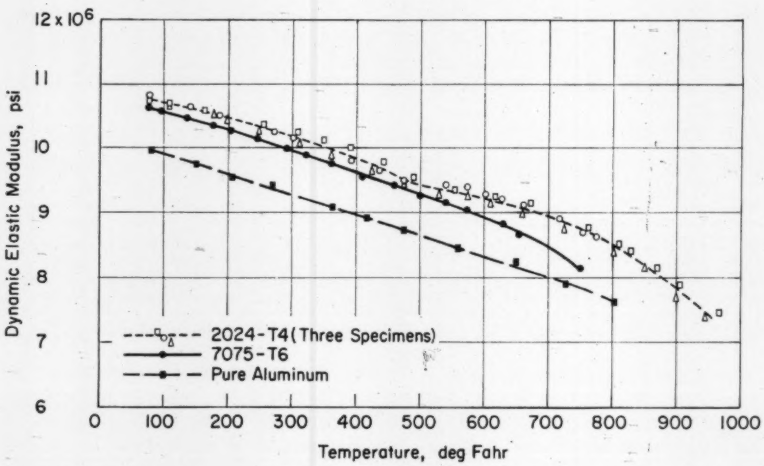


FIG. 2.—Effect of Temperature on the Dynamic Moduli of Pure Aluminum and Aluminum Alloys.

TEST RESULTS

Commercial Alloys and Pure Metals:

The effect of elevated temperature on the dynamic modulus of the commercial alloys and pure metals is presented graphically in Figs. 2 to 8. Insofar as practicable, related alloys and pure metals are grouped together.

Figure 2 shows the plot of dynamic elastic modulus versus temperature for pure aluminum and 2024-T4 and 7075 T-6 aluminum alloys. The effect of alloying additions in raising the modulus can be seen. The curve of modulus versus temperature for 2024-T4 shows an upward break in the curve at approximately 500 F. Since this inflection was unexpected, three specimens were tested in order to establish the validity of the results. The cause of the discontinuity is probably associated with aging, agglomeration, or solution of the precipitated phases, but further investigation of the phenomenon was beyond the scope of the original program.

In Fig. 3 the effect of temperature on the dynamic modulus of titanium and titanium alloys is shown. The room-temperature dynamic modulus of the iodide titanium, as obtained in this program, appears quite low when compared to values appearing in the literature. The low value is probably attributable to voids which could be seen in the microstructure. These voids were presumably due to gaps left between crystals during the growing process. The voids were not sufficient to lower the density of the material, which was found to be identical to that of the Ti-75A and the same as the handbook value for titanium. The modulus versus temperature curve for Ti-75A (which was from an early heat, about 1954) exhibits an inflection in the temperature range 250 to 400 F, which is probably associated with the high hydrogen content of the material used. The solubility of hydrogen in α-titanium below 257 F has been shown to be between 0.001 and 0.0029 per cent by weight (6). Metallographic examination confirmed the presence of two phases, α-titanium and hydride, at room temperature. The transformation temperature from the two-phase region to the single phase of α-titanium is shown by Hansen (6) to be about 275 F for a binary titanium-hydrogen alloy containing 0.03 per cent by weight hydrogen. The inflection in the curve is probably associated with the change from a two-phase to a single-phase material. The discontinuity in the modulus versus temperature curve for B-120 VCA is associated with aging of the specimen. Aging another specimen of the "as-received" material at 900 F for 48 hr raised the room-temperature modulus from 14.18×10^6 psi to 16.12×10^6 psi, and eliminated the inflection.

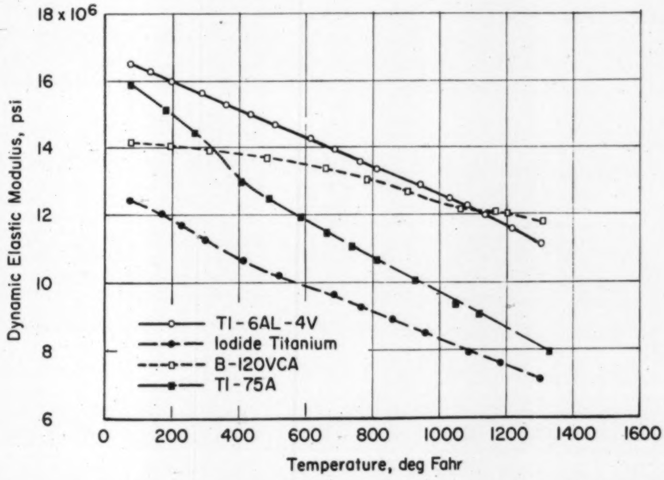
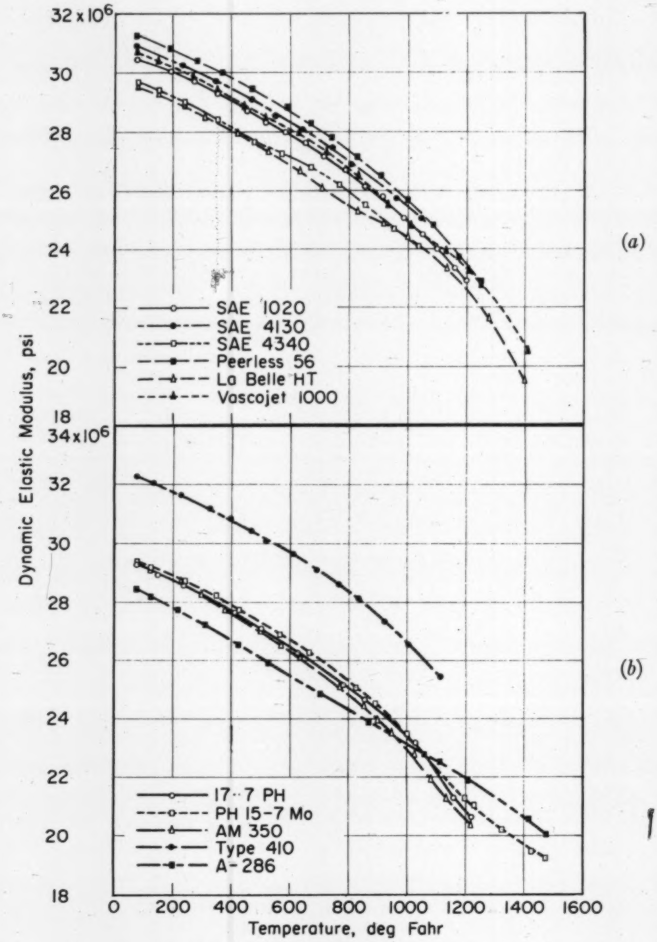


FIG. 3.—Effect of Temperature on the Dynamic Moduli of Iodide Titanium and Titanium Alloys.

The modulus versus temperature curves for one plain carbon and a group of low-alloy steels are shown in Fig. 4(*a*). The SAE 1020, SAE 4130, and Peerless

56 steels all exhibit smooth decreases in modulus with increasing temperature. These materials were stable over the temperature range investigated because: (1) the SAE 1020 was tested in the warm rolled condition; (2) the SAE 4130 was tempered at 850 F; (3) the Peerless 56 was tempered at 1000 F; and (4) under the continuous heating test conditions used in this program, tempering or aging effects were not realized at the maximum test temperature. The curves for SAE 4340, LaBelle HT, and Vascojet 1000 all exhibit inflections which are attributed to tempering effects: (1) The SAE 4340 which was tempered at 400 F exhibits a discontinuity at approximately 600 F; (2) the LaBelle HT, tempered at 500 F, shows a discontinuity at about 800 F; and (3) the Vascojet 1000, tem-



(*a*) Plain carbon and low-alloy steels.
(*b*) Stainless steels and an iron-base super alloy.

FIG. 4.—Effect of Temperature on the Dynamic Moduli of Steels.

pered at 1000 F, exhibits an inflection at about 1150 F.

Figure 4(*b*) shows the effect of elevated temperatures on the dynamic modulus of a group of stainless steels and the iron-base super alloy A 286. The type 410 stainless steel which was tempered at 1075 F displays a fairly smooth modulus versus temperature curve. The precipitation hardening stainless steels, 17-7PH, PH 15-7Mo, and AM 350, all show a tendency for the modulus versus temperature curve to break downward in the vicinity of the aging temperature. The A 286 alloy, which was aged at 1350 F, displays an almost linear decrease in modulus with increasing temperature up to 1400 F.

Dynamic modulus versus temperature curves for electrolytic nickel and a group of nickel-base alloys are shown in Figs. 5 and 6. The nickel-base alloys display a rather stable behavior over the range of temperatures. However, the modulus versus temperature curve for electrolytic nickel shows a pronounced hump over the temperature range from about 350 to 750 F. The reason for this behavior is not known but may be associated with magnetic properties.

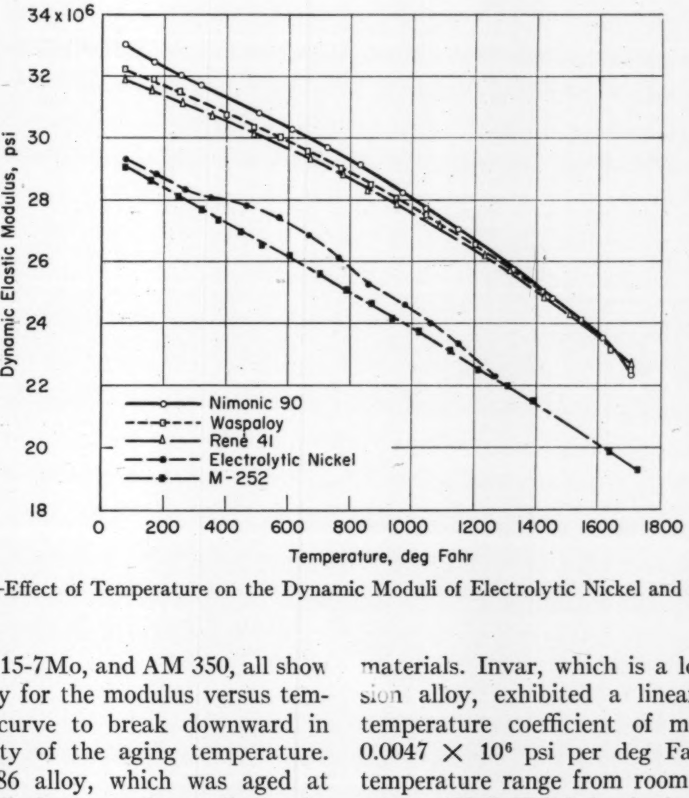
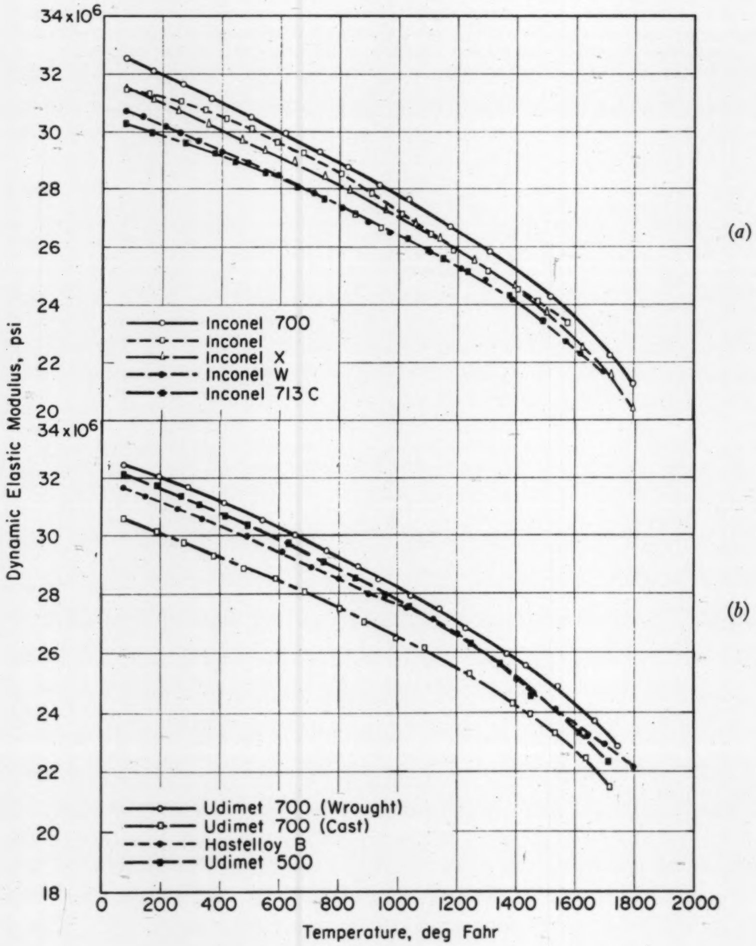


Fig. 5.—Effect of Temperature on the Dynamic Moduli of Electrolytic Nickel and Nickel-Base Alloys.

In Fig. 7 modulus versus temperature curves for three unrelated alloys are presented. These alloys did not fall logically into any of the other groups of materials. Invar, which is a low expansion alloy, exhibited a linear positive temperature coefficient of modulus of 0.0047 × 10^6 psi per deg Fahr in the temperature range from room temperature to 400 F. This behavior is attributed to the material's ferromagnetic behavior which disappears beginning in the range 325 to 400 F. At higher temperatures, the normal decreases of modulus with increasing temperature is evidenced. The beryllium-copper material investigated in this program was an experimental composition with a considerably lower beryllium content than the conventional alloy (refer to Table I). Heat treatment of this material had little effect on the dynamic modulus. S-816 was the only cobalt-base alloy investigated in this program. The dynamic modulus versus temperature behavior of unalloyed tungsten, molybdenum, and beryllium is shown in Fig. 8. The maximum test temperature for molybdenum and tungsten was limited to 1400 F because the specimen-holding screws were loosened by thermal expansion.



(*a*) Inconel.
(*b*) Udimet and Hastelloy alloys.

FIG. 6.—Effect of Temperature on the Dynamic Moduli of Nickel-Base Alloys.

Experimental Vanadium-Titanium Alloys:

In Fig. 9 it is seen that only a slight difference in the moduli of calcium-reduced vanadium and aluminothermic vanadium was found over the temperature range studied, even though the materials had two markedly different structures. The calcium-reduced vanadium had a cold-worked structure whereas the aluminothermic vanadium was recrystallized.

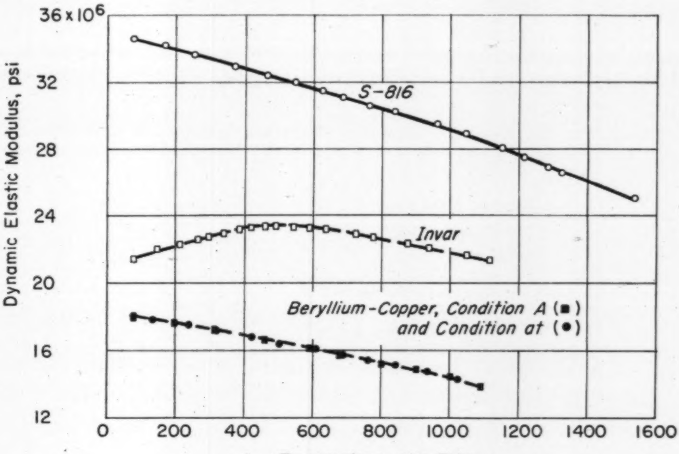


FIG. 7.—Effect of Temperature on the Dynamic Moduli of S-816, Invar, and Beryllium-Copper.

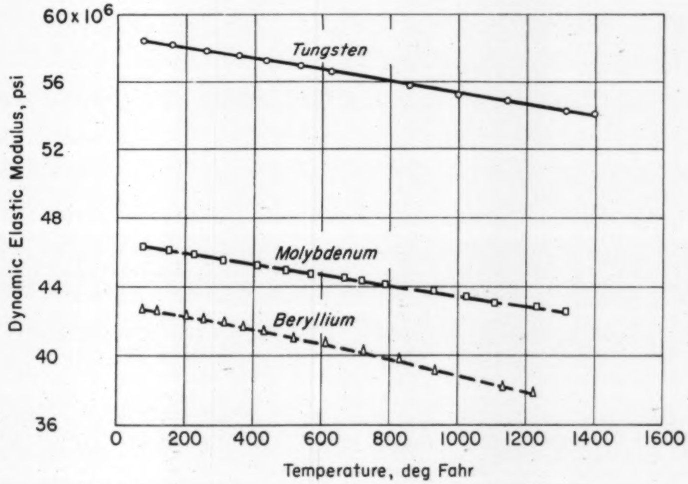


FIG. 8.—Effect of Temperature on the Dynamic Moduli of Unalloyed Tungsten, Molybdenum and Beryllium.

Figure 10 shows isothermal plots of modulus versus composition for room temperature, 300, 600, 900, and 1200 F, together with room-temperature modulus results of titanium-vanadium alloys determined by Graft et al (5). While increasing titanium contents lower the modulus both at room temperature and at elevated temperature, the alloy additions do not measurably affect the slope of the modulus versus temperature curves (Fig. 9). The modulus of iodide titanium was found to be considerably lower than that of the Ti-75A alloy over the entire temperature range. However, the decrease in modulus with increasing temperature was somewhat greater for Ti-75A.

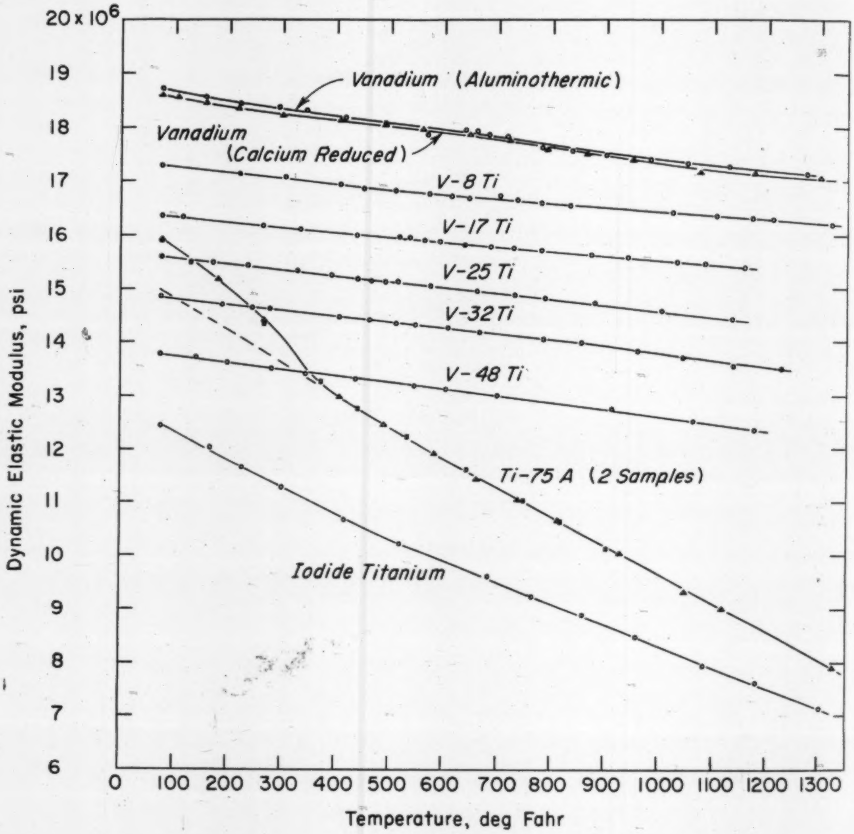


FIG. 9.—Effect of Temperature on the Dynamic Moduli of Vanadium, Titanium, and Vanadium-Titanium Alloys.

The relatively small decrease in modulus of vanadium with increasing temperature is characteristic of the other group 5A refractory metals—tantalum and columbium. Although titanium additions lower the modulus below that of pure vanadium, it is encouraging to note that vanadium-titanium alloys still exhibit a relatively small decrease in modulus with increasing temperature.

An interesting empirical correlation has been used by Cummings and Stulen (7), which relates the modulus of elasticity to density, atomic weight, and absolute melting point by the following equation:

$$E = k_0 \rho \frac{T_m}{A} \qquad \text{(4)}$$

where:

E = modulus of elasticity in psi × 10^{-6},
T_m = melting point temperature in degrees Rankine,

ρ = density in grams per cu cm,
A = atomic weight, and
k_0 = coefficient depending on lattice structure.

The empirically determined values of k_0 are: 0.059, face-centered cubic; 0.085, body-centered cubic; 0.051, hexagonal close-packed; and 0.102, hexagonal. Good agreement between the calculated and experimental values of E at room temperature have been found for Ag, Al, Au, Be, Ca, Co, Cr, Cu, Fe, Mg, Mo, Ni, Pb, Pd, Pt, Te, Ti, W, and Zr. Notable exceptions include Cb, Cd, Rh, Ru, Ta, and V. For these latter cases, if values of k_0 other than that of the known structure are substituted in the equation, surprisingly good agreement between experimental and calculated values of E are realized. For example, if k_0 for hexagonal close-packed rather than k_0 for body-centered cubic is substituted in the case of columbium, tantalum, and vanadium the calculated modulus values agree fairly closely with experimental values. No reason has been given for this fortuitous behavior.

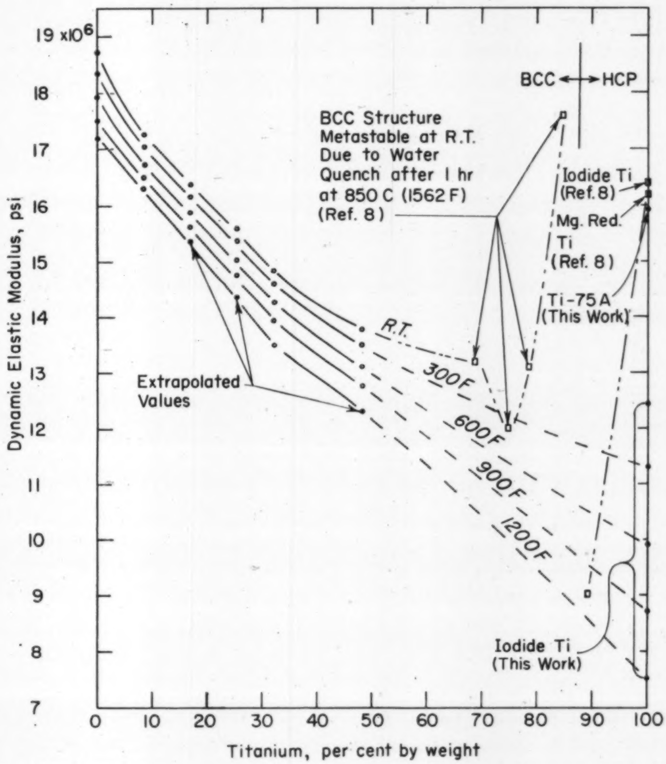


FIG. 10.—Isothermal Plots of Dynamic Modulus Versus Titanium Content.
Earlier work of Graft et al (5) on titanium-vanadium alloys shown for comparison purposes.

This empirical relationship has been satisfactorily applied to vanadium and vanadium-titanium alloys, the constant k_0 having been eliminated by determining the ratio of E (vanadium) to E (vanadium-titanium alloy) rather than calculating absolute values. This treatment assumes that k_0 does not appreciably change as a result of the titanium solid solution additions. The equation used is of the form:

$$\frac{E\ (\text{vanadium})}{E\ (\text{alloy})} = \left(\frac{T\rho}{A}\right)_{\text{vanadium}} \times \left(\frac{A}{T\rho}\right)_{\text{alloy}} \quad ..(5)$$

where A (alloy) $= (X_V A_V + X_{Ti} A_{Ti})$, X_V and X_{Ti} being the atom fractions of vanadium and titanium respectively. The values of temperature were taken from the vanadium-titanium phase diagram as reported by Hansen (6). The melting point of vanadium, as given by this diagram, is 3452 F, and the temperature values used for the various alloys were taken from the solidus curve. The calculated and experimentally determined ratios at room temperature are shown in Table III and it is seen that quite good agreement is realized between the two sets of values. The deviation between the measured and calculated ratios increases with increasing titanium content, the variation being about 1 per cent for V-8Ti, and about 5 per cent for V-48Ti.

TABLE III.—ROOM-TEMPERATURE PROPERTIES OF VANADIUM, TITANIUM, AND VANADIUM-TITANIUM ALLOYS.

Material	Vickers Hardness Number	Dynamic Modulus, psi	Density, lb per cu in.	E_V/E_{alloy}, measured	E_V/E_{alloy}, calculated	Deviation[a] of Measured and Calculated Ratios, per cent
Calcium-reduced vanadium	195	18.60 × 10^6	0.221	...	...	...
Aluminothermic vanadium	145	18.70	0.221	...	...	...
V-8Ti	223	17.28	0.213	1.082	1.070	1.11
V-17Ti	238	16.37	0.206	1.143	1.138	0.44
V-25Ti	278	15.59	0.200	1.200	1.182	1.50
V-32Ti	258	14.86	0.193	1.260	1.234	2.06
V-48Ti	250	13.79	0.188	1.359	1.286	5.37
Iodid titanium	84	12.45	0.163	...	...	...
Ti-75A	290	15.93	0.163	...	...	...
Ti-75A (extrapolated) from higher temperatures	...	15.05	0.163	...	...	...

[a] Per cent deviation defined as $\left(\frac{\text{measured ratio} - \text{calculated ratio}}{\text{measured ratio}}\right) \times 100$.

Since the modulus versus temperature curves for the vanadium and vanadium-titanium alloys are nearly parallel, the experimental ratio of E_V to E_{alloy} for a given alloy at a particular elevated temperature is quite close to the ratio at room temperature. If the density of pure vanadium and the vanadium-titanium alloys changed proportionally with temperature, then ratios at elevated temperatures calculated using Eq 5 could be expected to agree fairly closely with the corresponding experimentally determined ratios.

Therefore, for this particular binary system it is possible to determine with an estimated 90 per cent accuracy the modulus of elasticity as a function of temperature for any vanadium-titanium alloy up to 48 per cent by weight titanium, knowing only the variation of modulus of pure vanadium with temperature, and pertinent phase diagram data. If the density of the alloys at room temperature could not be readily measured, reasonable estimations could be made. In addition, the assumption would have to be made that the density of the pure vanadium and that of the alloys changed proportionally with temperature.

While application of this empirical correlation has proven quite successful in the case of binary vanadium-titanium alloys, it is not to be implied that such good agreement will be found in all binary systems. However, another example of a binary system showing fair agreement between measured ratios and calculated ratios is the Cr-Fe system (8). An addition of 40 per cent by weight of iron to chromium lowers the modulus of elasticity at room temperature from 42×10^6 psi to 33.5×10^6 psi, giving a ratio of E_{Cr}/E_{alloy} of 1.254. The calculated ratio, using Eq 5, is 1.177.

SUMMARY AND CONCLUSIONS

1. This investigation demonstrated that the elastic moduli of metals may be determined dynamically at elevated temperatures by using longitudinal resonant frequency measurements. This method was employed in establishing the elevated temperature dynamic moduli of 45 metals and alloys reported herein. A room-temperature comparison of dynamic and static moduli has been presented for most of the materials investigated.

2. This simple and straightforward method of determining dynamic modulus requires only one specimen for a complete modulus-temperature plot. It has provided reproducible modulus data which, in some cases, indicate a sensitivity to structural changes taking place in a material.

3. The temperature-dependence of the elastic modulus in a series of experimental vanadium-titanium alloys has been determined. Additions of titanium to vanadium (up to 48 per cent by weight titanium) lowered the modulus of binary alloys below that of pure vanadium, both at room temperature and at elevated temperatures. The titanium additions did not, however, affect the slope of the modulus versus temperature curve. An empirical correlation relating modulus to density, atomic weight, and the absolute melting point was applied successfully to the vanadium-titanium alloys. The calculated ratios of E (vanadium) to E (alloy) at room temperature were found to agree closely with the corresponding ratios determined using experimental modulus results.

Acknowledgments:

The authors are very grateful to Mr. E. H. Beutel for his assistance in equipment construction, and to Messrs. D. M. Forney, Jr. and W. J. Trapp for their support and encouragement.

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DISCUSSION

Mr. Sam Spinner.[1]—The authors indicated that the upper limit at which they stopped making determinations was due to some kind of experimental failure. However, I noticed that with certain materials they were able to go further than others, suggesting that some materials actually continue to give responses where other metals stopped at an earlier stage. Was that correct?

Mr. K. D. Shimmin (*author*).—Actually on the materials in which oxidation is not a problem, materials that are usually used in an oxidizing atmosphere anyway, the furnace is opened for a short time and the screws are tightened before they become loose; then we were able to continue on to higher temperatures.

Mr. Spinner.—Certain vanadium alloys went to around 1400 F and others stopped about 200 F lower.

Mr. Shimmin.—This is true. The vanadium-titanium alloys were run in an inert atmosphere, and in some cases we did get a loosening of the fixture, the specimen fell out of position, and the test had to be discontinued. In some cases this did not happen so we were able to go up to a higher temperature.

Mr. Spinner.—Is there any possibility that at a high enough temperature the specimens themselves failed to respond due to internal friction at these (elevated) temperatures?

Mr. Shimmin.—Our limit is probably 1800 F. At 1800 F, no matter what we do, we tend to lose the signal and that is the upper temperature limit.

Mr. Spinner.—After you measured at elevated temperature and then came back to room temperature, was any change in frequency detected? In other words, did the heating itself change the specimens significantly?

Mr. Shimmin.—We did not mention it in the paper, but in some cases we actually took readings during heating and during cooling. Tests on some specimens show a difference due to the aging, or other metallurgical changes which take place. In these cases a permanent change in room temperature resonant frequency is observed.

Mr. Donald R. Erb.[2]—What is the accuracy of reproducibility between two or possibly three specimens of the same material at a given temperature using this experimental equipment?

Mr. Shimmin.—That would depend to a certain extent on the material. The curve for 2024 aluminum is actually made up of the results of three tests, and the points are shown on this curve. In this case there is a discontinuity in the curve since heating rates are only nominal value and there is some effect on the scatter in the curve due to longer or shorter times in reaching the temperature where the aging takes place. On a stable material we can come back down to room temperature and the next day get exactly the same value.

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[2] Engineering Manager, Instron Engineering Corp., Canton, Mass.

THE APPLICATION OF RABOTNOV'S CREEP PARAMETER*

By Robert M. Goldhoff[1]

Many machine components, though not creep-limited in design, are subject to deformation limits in specified design times. The design procedure usually requires knowledge of the creep characteristics of materials under consideration. This is normally obtained in the familiar creep apparatus using uniaxial tension and can be a highly time-consuming process. Methods whereby the minimum creep data can be used to extrapolate to longer times are needed. One such method is that proposed by Rabotnov (**1**),[2] and its application is the subject of this paper.

Rabotnov's Concept

Several basic methods for correlating creep data have been proposed, and Manson (**2**) has discussed these briefly. The "K factor" method of Shepard (**3**) attempts to reduce a family of isothermal creep curves at various stresses to a single curve by simply multiplying the time scale for each curve by a constant. Applications of this method have shown tendencies for large errors and not wholly satisfactory results. The "θ factor" method proposed by Dorn (**4**) suggests that a family of isostress creep curves can be brought into coincidence by plotting strain versus a temperature-modified time. This process can be carried one step further by recognizing the time-temperature parameter as a function of stress. Then the familiar master curve of stress versus time-temperature parameter for constant strain results (Fig. 1). This method is subject to the numerous assumptions inherent in the time-temperature parameter. Most of these have been thoroughly discussed previously, the chief objection being the unknown factor of metallurgical instability in long times and at excessive temperatures.

Rabotnov's method derives from Soderberg's (**5**) theory in which creep is a function of plastic strain (ϵ_p), stress (σ), and time (t):

$$\phi(\epsilon_p, \sigma, t) = 0 \quad \text{(1)}$$

The variant proposed is:

$$\sigma = f(\epsilon, t) \quad \text{(2)}$$

where ϵ is now total strain.

To facilitate computation, the conventional family of creep curves is redrawn in the coordinates of stress and strain for definite values of time as shown in Fig. 2(*a*). This procedure has an immediate interest for those concerned with design, because the computations for structural parts normally involve the determination of stresses for fixed values of strain and time.

As in the previous methods discussed, Rabotnov found that the isochronous creep curves are similar, that is, they can be obtained from one curve by multiplying its ordinates by a constant which is a function of time. Returning to Eq 2, it is seen that the dependence of stress on deformation and time can be treated as a product of two functions, which in turn are functions of deformation and time only:

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* Presented at the Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] Applied Research Metallurgist, Large Steam Turbine-Generator Dept., Materials and Processes Laboratory, General Electric Co., Schenectady, N. Y.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

$$\sigma = \phi(\epsilon)\Sigma(t) \qquad \text{(3)}$$

Now if it is assumed that $\Sigma(o) = 1$, then $\phi(\epsilon)$ will describe the stress-strain diagram approximated by the short-time tension test at a like temperature. This is illustrated in Fig. 2(*b*).

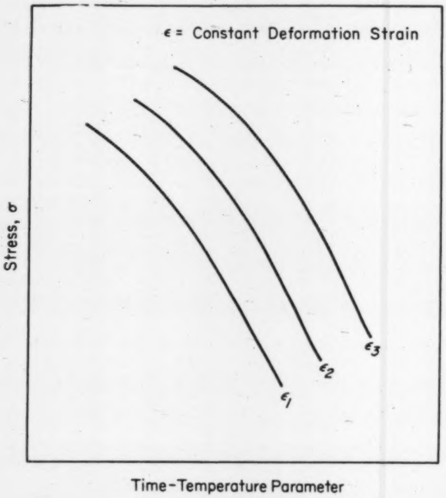

FIG. 1.—Method of Presenting Creep Data by Plot of Stress Versus Time-Temperature Parameter.

Upon examination of many isochronous families of creep curves, Rabotnov proposed the following equation:

$$\sigma = \phi(\epsilon)\left[\frac{1}{1 + G(t)}\right] \qquad \text{(4)}$$

where

$$G(t) = \left(\frac{\beta}{1 - \alpha}\right) t^{(1-\alpha)} \qquad \text{(5)}$$

In Eq 5, α is said to fluctuate about 0.7, while β is highly dependent on the material being investigated. For application purposes, the form

$$\Sigma(t) = \frac{1}{1 + at^b} \qquad \text{(6)}$$

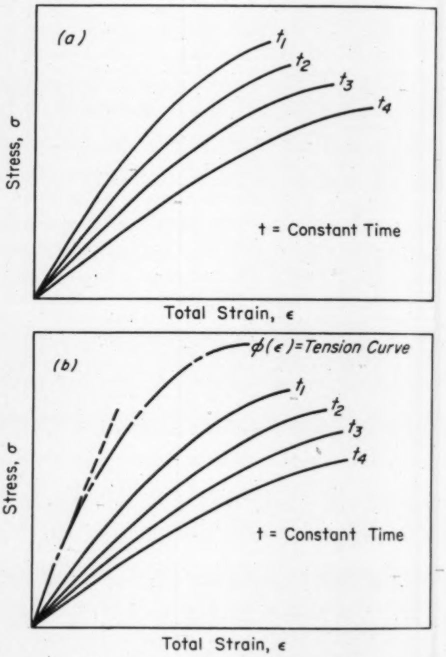

FIG. 2.—Method of Presenting Creep Data by Plotting Isochronous Creep Curves.

can be used, and this leads to the final expression:

$$\phi(\epsilon) = \sigma(1 + at^b) \qquad \text{(7)}$$

Turner and Blomquist (6) studied this method with reference to data on an alloy of 24S-T aluminum. The actual mechanics of the parameter application are well outlined in that work. If *b* can be assumed from Rabotnov's work, then values for both $\phi(\epsilon)$ and *a*, which are dependent on strain, must be known if the method is to be used for generating any one of a particular family of isochronous creep curves. If the tension stress-strain curve is available, $\phi(\epsilon)$ is assumed to be the value of σ on this curve corresponding to a pertinent strain level. The value of a for the same strain can then be calculated using Eq 7 provided one other isochronous creep curve is known. Armed with these values, it is now theoretically possible to calculate the creep stress for any time leading to the given deformation at the temperature being studied. In the absence of the tension stress-strain curve, a pair of simultaneous equations of the form of Eq 7 can be developed from any two isochronous curves, and these will yield values of both $\phi(\epsilon)$ and a. It is possible in this way to generate the master curve $\phi(\epsilon)$, and it should closely approximate the tension stress-strain curve. The remainder of this paper is devoted to testing the accuracy of this method.

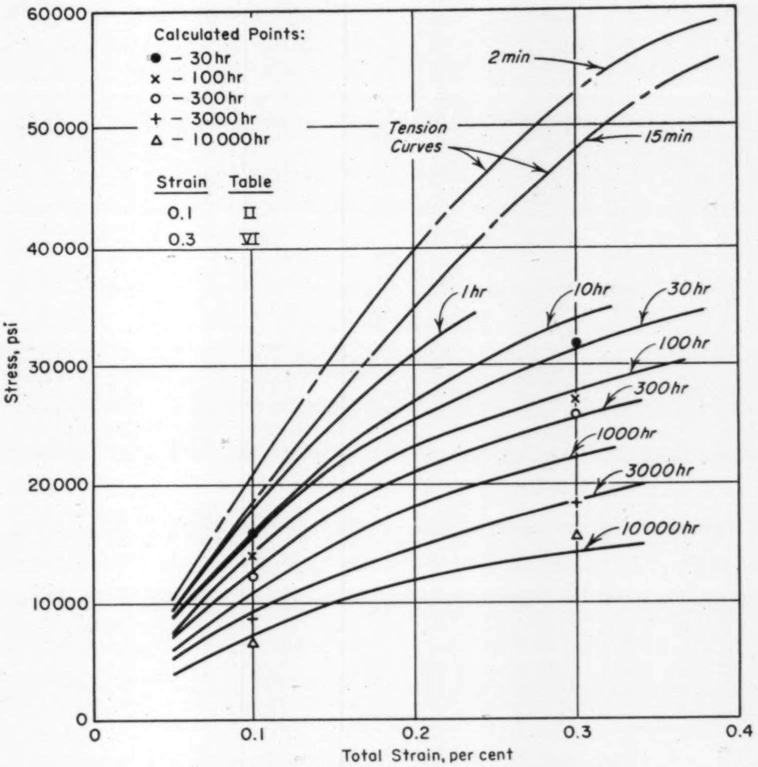

FIG. 3.—Isochronous Creep Curves, Cr-Mo-V Steel, 1050 F.

APPLICATION OF THE PARAMETER

The following questions are to be considered:

1. Can creep data from varying types of materials be correlated by this method?
2. If so, how sensitive is the method with respect to the correlating functions?
3. Is the short-time tension curve reliable as a master curve for the correlation?

4. What is the potential of this method for design use?

Choice of Data:

Data from the following types of materials from this laboratory as well as those obtained from a literature search were studied:

(*a*) a heat-treatable ferritic steel (1 Cr, 1 Mo, $\frac{1}{4}$ V) tested at 1050 F,[3]

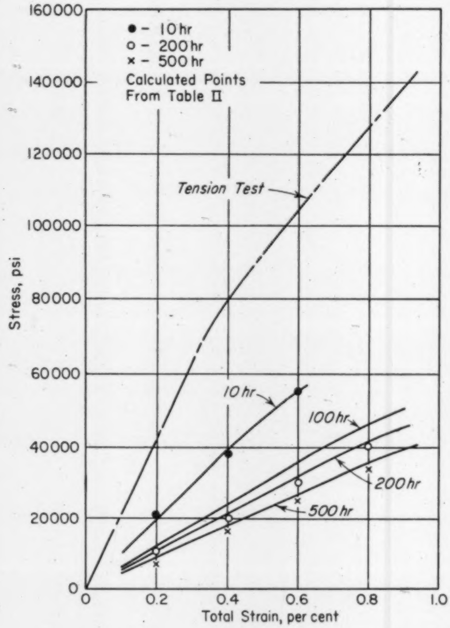

FIG. 4.—Isochronous Creep Curves, Crucible 422 Steel, 1000 F.

(*b*) a 12 per cent Cr alloy (Type Crucible 422) tested at 800, 1000, and 1200 F,[4]

(*c*) an austenitic steel (Type A-286) tested at 1200 and 1300 F,[5]

(*d*) a light alloy typified by magnesium tested as sheet at 300 F and as extrusions at 200 F,[6]

(*e*) plastics as represented by canvas laminate and nylon tested at 77 F,[7] and

(*f*) a precipitation-hardening steel alloy (17-7 PH type, 1050 TH condition) tested at 900 F.[8]

Typical isochronous data from *a*, *b*, *e*, and *f* are shown in Figs. 3 to 6. Short-time tension data are included where available. Complete stress-strain data are given in Table I.

Fit of Data:

Examination of Eq 7 shows that in the absence of a tension curve, the data fit depends on the three quantities $\phi(\epsilon)$, b, and a. Each set of data was analyzed to find the best values of these quantities for predetermined strain levels. To accomplish this, a least-squares method of predicting the coefficients was chosen for machine programming. Use was made of the fact that the n simultaneous equations developed are linear in $\phi(\epsilon)$ and a, although nonlinear in b. Hence, by assuming a value for b, the corresponding solutions for $\phi(\epsilon)$ and a can be obtained. By incrementing b and reducing the sum of the squares of deviations between actual and predicted stress values to a minimum, the best combination of $\phi(\epsilon)$, a, and b were determined. These are compiled in Table I. The best values of constants appear to be strain-, temperature-, and material-dependent. Where tension data are available, in general, the best values of $\phi(\epsilon)$ do not correspond with the values taken therefrom. However, it was noted that for the values of $\phi(\epsilon)$ which most closely correspond to the tension data the solution for b is nearly always in the vicinity of 0.30 to 0.33. Hence, it appears that Rabotnov's criteria established in accordance with Eqs 4 and 5 are valid. The best correlation will be sacrificed in order to incorporate the usefulness of deriving $\phi(\epsilon)$ from the tension curve and assuming a constant value of exponent b independent of strain, temperature, and material. Nevertheless, the usefulness of the method is dependent on the degree and trend of deviations between predicted and actual values.

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[3] Materials and Processes Laboratory, General Electric Co., Schenectady, N. Y.

[4] Am. Soc. Metals Handbook Committee on Wrought and Heat Resistant Steels, P. Payson.

[5] Am. Soc. Metals Handbook Committee on Wrought and Heat Resistant Steels, E. E. Reynolds.

[6] H. Baker, "Tensile and Creep Properties at Elevated Temperatures of Some Magnesium-Base Wrought Alloys," *Proceedings*, Am. Soc. Test Mats., Vol. 54, p. 1068 (1954).

[7] W. N. Findley and D. B. Peterson, "Prediction of Long-Time Creep with Ten-Year Data on Four Plastic Laminates," *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 841 (1958); J. Marin, A. C. Webber, and G. F. Weissmann, "Creep-Time Relations for Nylon in Tension, Compression, Bending, and Torsion," *Ibid*, Vol. 54, p. 1313 (1954).

[8] Am. Soc. Metals Handbook Committee on Wrought Heat Resistant Steels, W. C. Clarke.

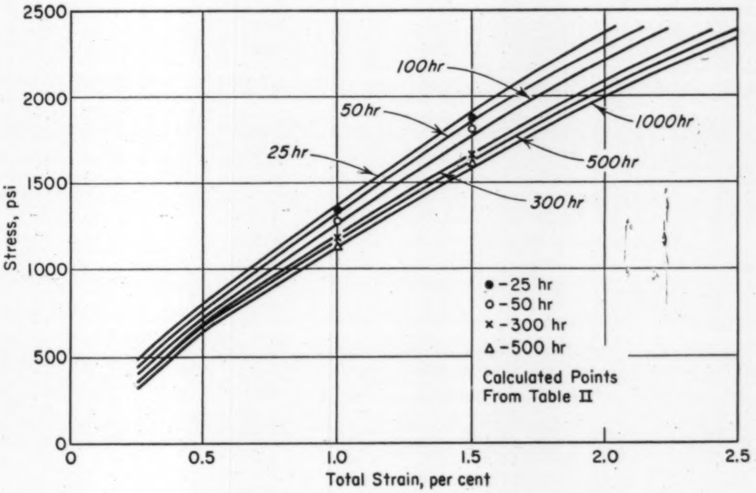

FIG. 5.—Isochronous Creep Curves, Nylon, 77 F.

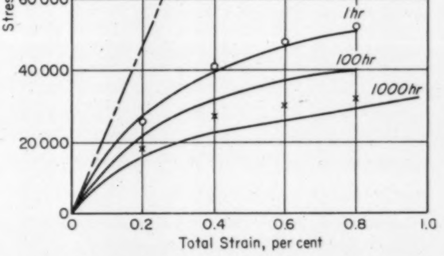

FIG. 6.—Isochronous Creep Curves, 17-7 PH (TH 1050), 900 F.

To check the accuracy of interpolation and extrapolation of the isochronous creep data by this method, the value of $b = 0.33$ was used for the metallic alloys (excluding 17-7 PH) and $b = 0.10$ for the plastic materials and for 17-7 PH. Where tension data were available, values of $\phi(\epsilon)$ for selected strains were observed, and, in conjunction with a median isochronous curve, the remaining curves were predicted. In the absence of tension data, two widely separated isochronous curves were chosen and the master curve generated. Again, the remaining curves were predicted. Comparison between predicted and actual stresses, along with percentage errors in prediction, are shown in Table II. Some graphical comparisons of actual curves and estimated points are

TABLE I.—RESULTS OF STATISTICAL COMPUTATION OF CONSTANTS IN RABOTNOV'S EQUATION, $\phi(\epsilon) = \sigma(1 + at^b)$.

Alloy	Total Strain, per cent	$\phi(\epsilon)$	b	a	$\phi(\epsilon)$ (Tensile Data)	b	a
Low Alloy Steel (1050 F)	0.05	9 500	0.40	0.037	10 250	0.32	0.08
	0.10	18 597	0.38	0.049	20 500	0.30	0.11
	0.20	33 152	0.32	0.094	41 250	>0.30	...
	0.30	38 657	0.34	0.072	53 000	>0.30	...
Crucible 422 (800 F)	0.20	46 185	0.44	0.038	55 000	0.30	0.12
	0.40	92 592	0.42	0.041	96 300	0.38	0.056
	0.60	120 600	0.54	0.012	125 000	0.42	0.028
Crucible 422 (1000 F)	0.20	51 102	0.28	0.88	49 900	0.30	0.63
	0.40	113 094	0.28	1.00	79 400	0.32	0.53
	0.60	181 906	0.24	1.35	103 600	0.30	0.48
	0.80	60 483	0.52	0.030	128 500	>0.30	...
Crucible 422 (1200 F)	0.20	17 585	0.50	0.086	28 700	0.30	0.56
	0.40	44 305	0.30	0.688	41 870	0.30	0.68
	0.60	69 184	0.28	1.04	47 130	0.32	0.46
A-286 (1200 F)	0.50	87 205	0.28	0.195	83 600	0.30	0.16
	1.00	92 846	0.28	0.176	...	...	...
	2.00	98 613	0.26	0.206	...	...	...
	3.00	103 663	0.24	0.251	...	...	...
A-286 (1300 F)	0.50	96 890	0.28	0.557	81 600	0.32	0.352
	1.00	101 190	0.28	0.484	...	...	...
	2.00	93 760	0.30	0.365	...	...	...
	3.00	95 555	0.30	0.367	...	...	...
Mg Sheet (300 F)	0.10	4 720	0.64	0.176	...	...	...
	0.50	13 426	0.42	0.331	...	...	...
	1.00	13 166	0.50	0.149	...	...	...
Mg Extrusion (200 F)	0.10	6 843	0.34	0.120	...	...	...
	0.50	17 054	0.32	0.103	...	...	...
	1.00	22 300	0.24	0.180	...	...	...
Canvas Laminate (77 F)	0.20	2 070	0.14	0.460	...	...	...
	0.50	3 340	0.16	0.151	...	...	...
	1.00	5 816	0.12	0.220	...	...	...
	1.50	7 334	0.12	0.180	...	...	...
Nylon (tension) (77 F)	0.50	995	0.18	0.153	...	...	...
	1.00	1 998	0.14	0.298	...	...	...
	1.50	2 736	0.14	0.281	...	...	...
Armco 17-7 PH (950 F)	0.20	30 973	0.32	0.097	43 000	0.16	0.497
	0.40	41 309	0.38	0.059	85 700	0.10	1.03
	0.60	47 927	0.42	0.046	102 000	0.12	0.80
	0.80	54 876	0.38	0.064	109 000	0.10	1.07

shown in Figs. 3 to 6. The predictions are quite good, with maximum errors being about ±5 to 8 per cent. Exceptions to this occur with the low-alloy steel at the 0.2 and 0.3 per cent strain levels, the magnesium sheet alloy at 0.1 per cent strain, and the 17-7 PH alloy. For the low-alloy steel particularly, the errors have a fairly definite trend. The following section inquires into this matter.

Miscellaneous Considerations:

A well-known effect is that of strain rate in tension testing. Generally, increasing the strain rate causes displacement of stress to higher levels for the same strain. This effect is highly dependent on the temperature. Where tension data are shown in this report, they are for test times of about 2 min to attain the total strain shown. Any large difference due to strain rate could affect the use of the tension data for a master curve in this correlation. Tension tests were performed on the low-alloy steel at 1050 F in times ranging from 30 sec to 15 min up to 1.0 per cent strain. The curves for 2 and 15 min are shown in Fig. 3. From this work, it appears that times between 30 sec and 4 min gave essentially identical curves. In the useful creep temperature range, this should serve for a short-time tension curve for most materials.

A valid consideration is what effect could the use of various isochronous curves in conjunction with a tension curve have on predicted results? Again, for the low-alloy steel at 0.1 per cent strain the values of a and the 10,000-hr strength were calculated using each curve in turn. The results are shown in Table III. It is apparent that the use of short-time curves will lead to appreciable errors in long-time prediction for this alloy at 1050 F. Curves of 100 hr and above will predict the 10,000-hr strength accurately but in so doing will compromise the shorter-time predictions. This may be a function of the particular set of data, because reference to Table I shows that this does not seem to be a general trend for the data treated. Further, the errors for estimating this set of data increase with the strain level, which is not a general trend for the various data considered.

This leads to an inquiry as to the sensitivity of the method to the value of exponent b and also to the relative merit of developing a master curve from the creep data, as opposed to the use of the tension curve. To this end, the data of the low-alloy steel and nylon were retreated using other values of b. For the steel, b was changed from 0.33 to 0.30, while for the nylon a new value of 0.30 for b was used. The results of this analysis are shown in Table IV. By comparison with Table I, it appears that the plastic is quite insensitive to large changes in b, whereas the low-alloy steel shows considerable improvement for a small change in b. Nevertheless, in the latter case the error increase with strain level persists. In addition, the errors do not appear to be random.

Next, the master curve is developed for the low-alloy steel data by using the 10-hr and 1000-hr creep curves and both values of b used previously, that is, 0.30 and 0.33. Table V shows this result. For this set of data, the calculated master curve deviates to a large degree from the tension curve as the strain increases. As might be expected, it is closer to the values predicted by the least-squares analyses. Finally, the creep curves were estimated using the apparently most ideal case in which the master curve is derived from creep data and the value of b is 0.30. The comarison is shown in Table VI, and the improvement is noteworthy. Even though the error in extrapolation to 10,000 hr is of the same order of magnitude (5 to 10 per cent), the predictions appear to have random errors. The values from Table VI for 0.3 per cent strain are plotted in Fig. 3, and the comparison between actual and calculated values is seen to be good.

TABLE II.—COMPARISON OF ESTIMATED AND ACTUAL CREEP STRESSES BASED ON THE USE OF RABOTNOV'S PARAMETER.

Alloy	Strain, per cent	Time, hr	Calculated Stress, psi	Actual Stress, psi	Error, $\sigma_{calc}-\sigma_{act}/\sigma_{act}$, per cent
Low-Alloy Cr-Mo-V, 1050 F (based on tension curve and 1000-hr curve, b = 0.33)	0.05	1	9 520	9 200	+3.49
		10	8 860	8 700	+1.84
		30	8 400	8 300	+1.20
		100	7 700	7 700	0
		300	6 960	6 900	+0.87
		3 000	5 070	5 100	−0.59
		10 000	4 060	3 800	+6.85
	0.10	1	18 900	17 700	+6.77
		10	17 200	16 600	+3.60
		30	16 150	15 800	+2.17
		100	14 600	14 600	0
		300	12 900	13 200	−2.27
		3 000	9 050	9 200	−1.63
		10 000	7 050	7 200	−2.08
	0.20	1	33 800	31 100	+8.7
		10	31 900	27 100	+17.7
		30	29 300	25 500	+14.9
		100	25 700	23 200	+10.7
		300	22 300	21 300	+4.69
		3 000	14 750	14 800	−0.33
		10 000	11 250	11 900	−5.46
	0.30	10	40 700	33 900	+20.0
		30	37 100	31 200	+18.9
		100	32 300	27 800	+16.2
		300	27 500	25 500	+7.84
		3 000	17 700	18 700	−5.35
		10 000	13 400	14 300	−6.30
Crucible 422, 800 F (based on tension curve and 100-hr curve, b = 0.33)	0.20	10	44 400	41 800	+6.2
		200	33 400	32 500	+2.8
		500	29 200	29 300	−0.3
	0.40	10	83 500	83 500	0
		200	68 100	66 500	+2.4
		500	61 600	59 500	+3.5
	0.60	10	115 400	115 600	−0.17
		200	102 000	98 750	+3.3
		500	95 800	89 750	+7.0
Crucible 422, 1000 F (based on tension curve and 100-hr curve, b = 0.33)	0.2	10	19 500	19 100	+2.1
		200	10 200	10 500	−2.9
		500	8 050	8 490	−5.2
	0.4	10	38 100	39 100	−2.5
		200	20 200	21 000	−3.8
		500	15 900	16 900	−5.9
	0.6	10	54 700	54 700	0
		200	30 200	31 200	−3.2
		500	24 100	26 200	−8.0
	0.8	200	39 900	39 100	+2.0
		500	32 000	34 300	−6.7

TABLE II.—*Continued.*

Alloy	Strain, per cent	Time, hr	Calculated Stress, psi	Actual Stress, psi	Error, $\sigma_{calc}-\sigma_{act}/\sigma_{act}$, per cent
Crucible 422, 1200 F (based on tension curve and 100-hr curve, $b = 0.33$)	0.20	10	14 900	13 810	+8.0
		200	8 200	7 800	+5.1
		500	6 540	6 050	+8.3
	0.4	10	19 200	18 700	+2.7
		200	10 000	10 000	. . .
		500	7 850	8 200	−4.3
	0.6	200	12 300	12 400	−0.8
		500	9 730	10 000	−2.7
A-286 1200 F (based on tension curve and 1-hr curve, $b = 0.33$)	0.5	10	63 300	64 000	−1.1
		100	49 500	50 800	−2.56
		1 000	33 700	37 200	−9.4
(based on 1-hr and 1000-hr curves, $b = 0.33$)	1.0	10	70 600	70 000	+0.86
		100	58 000	56 400	+2.84
	2.0	10	73 500	72 000	+2.08
		100	60 600	58 400	+3.77
	3.0	10	74 600	72 400	+3.04
		100	61 500	58 800	+4.60
A-286 1300 F	0.5	10	46 200	50 000	−7.60
		100	32 400	33 900	−3.96
	1.0	10	53 400	53 500	−0.187
		100	37 000	37 900	−2.37
	2.0	10	54 000	55 500	−2.70
		100	38 400	39 200	−2.04
	3.0	10	56 100	55 600	+0.90
		100	39 600	38 600	+2.59
Mg Sheet, 300 F (based on 1-hr and 100-hr curves, $b = 0.33$)	0.10	3	2 980	3 700	−19.4
		10	2 160	2 600	−16.9
		30	15 800	18 000	−12.2
	0.50	3	8 600	8 800	−2.27
		10	6 900	7 200	−4.16
		30	5 480	5 600	−2.14
	1.00	3	10 100	10 400	−2.88
		10	8 400	9 000	−6.67
		30	6 860	7 200	−4.72
Mg Extrusion, 200 F (based on 1-hr and 100-hr curves, $b = 0.33$)	0.10	3	5 800	6 100	−4.92
		10	5 420	5 800	−6.55
		30	4 970	5 000	−0.60
	0.50	3	14 900	15 000	−0.67
		10	14 100	14 000	+0.71
		30	13 100	13 000	+0.77
		300	10 400	10 400	0
	1.00	3	18 200	18 000	+1.11
		10	17 100	17 000	+0.59
		30	15 900	16 000	−0.62
		300	12 450	13 100	−4.80

Continued on p. 916

TABLE II.—*Concluded.*

Alloy	Strain, per cent	Time, hr	Calculated Stress, psi	Actual Stress, psi	Error, $\sigma_{calc}-\sigma_{act}/\sigma_{act}$, per cent
Canvas Laminate, 77 F (based on 1-hr and 1000-hr curves, $b = 0.10$)	0.20	0.1	1 415	1 570	−9.85
		10	1 240	1 250	−0.80
		100	1 085	1 120	−3.12
		10 000	800	770	+3.90
	0.5	0.1	2 920	3 030	−3.63
		10	2 720	2 730	−0.37
		100	2 540	2 530	+0.40
		10 000	2 100	2 000	+5.0
	1.00	0.1	4 800	4 980	−3.62
		10	4 500	4 520	−0.44
		100	4 190	4 210	−0.43
		10 000	3 520	3 500	+0.57
	1.50	0.1	6 260	6 430	−2.65
		10	5 930	5 940	−0.17
		100	5 580	5 580	0
		10 000	4 800	4 750	+1.05
Nylon 77 F (based on 100-hr and 1000-hr curves, $b = 0.10$)	0.50	25	780	790	−1.27
		50	755	755	0
		300	695	700	−0.72
		500	680	680	0
	1.00	25	1 340	1 370	−2.20
		50	1 300	1 320	−1.51
		300	1 195	1 200	−0.42
		500	1 160	1 130	+2.66
	1.50	25	1 880	1 910	−1.57
		50	1 830	1 850	−1.08
		300	1 690	1 670	+1.20
		500	1 640	1 630	+0.61
17-7 PH (TH-1050) 900 F (based on tension curve and 100-hr curve, $b = 0.10$)	0.2	1	26 600	27 700	−3.9
		1 000	19 300	16 400	+17.7
	0.4	1	40 400	39 000	+3.6
		1 000	26 400	22 800	+15.8
	0.6	1	47 600	45 800	+3.93
		1 000	31 100	26 000	+19.6
	0.8	1	52 300	51 600	+1.35
		1 000	34 400	29 100	+18.2

In a number of creep laboratories, the method of testing leads more easily to a representation of the data in terms of time versus plastic strain rather than total strain. Curiosity led to the examination of the low-alloy steel data based on plastic strain. Elastic strain was simply subtracted from the tension curve and plotted along with plastic-strain creep data. The results of the standard analysis using such data are given in Table VII. Comparison with its counterpart reveals somewhat larger errors. It is likely that this procedure should be avoided.

TABLE III.—EFFECT OF VARIATION OF CONSTANT *a* ON PREDICTION OF 10,000-HR DATA FOR LOW-ALLOY STEEL.

Time Curve Used, hr	a ($b = 0.33$)	Estimated 10,000-hr Strength, psi	Actual 10,000-hr Strength, psi	Error, per cent
1	0.157	4720	7200	−34.4
10	0.116	5900	7200	−18.0
30	0.097	6700	7200	−6.95
100	0.088	7100	7200	−1.39
300	0.086	7250	7200	+0.70
1000	0.087	7200	7200	0
3000	0.087	7200	7200	0

TABLE IV.—COMPARISON OF CREEP STRESSES FOR DETERMINATION OF SENSITIVITY TO CHANGES IN CONSTANT *b*.

Alloy	Strain, per cent	Time, hr	Calculated Stress, psi	Actual Stress, psi	Error $\sigma_{calc}-\sigma_{act}/\sigma_{act}$, per cent
Low-Alloy Cr-Mo-V, 1050 F (based on tension curve and 1000-hr curve, $b = 0.30$)	0.05	10	8 700	8 700	0
		30	8 200	8 300	−1.2
		100	7 550	7 700	−1.95
		300	6 850	6 900	−0.73
		3 000	5 120	5 100	+0.39
		10 000	4 200	3 800	+10.5
	0.30	10	39 100	33 900	+15.0
		30	35 400	31 200	+13.4
		100	31 000	27 800	+11.5
		300	26 700	25 500	+4.7
		3 000	17 800	18 700	−4.8
		10 000	13 900	14 300	−2.8
Nylon, 77 F (based on 100-hr and 1000-hr curves, $b = 0.30$)	0.5	25	760	790	−3.7
		50	745	755	−1.32
		300	695	700	−0.66
		500	680	680	0
	1.00	25	1 320	1 370	−3.65
		50	1 290	1 320	−2.27
		300	1 210	1 200	+0.83
		500	1 180	1 130	+4.40
	1.50	25	1 850	1 910	−3.14
		50	1 810	1 850	−2.16
		300	1 700	1 670	+1.80
		500	1 660	1 630	+1.84

THE DESIGN PROBLEM

The specific design problem will be altered by the case considered. In general, the problem is one of extrapolation using the minimun data. Ideally, as shown in the time-temperature parameter evaluation of Fig. 1, a minimum of three tests, chosen judiciously with respect to stress and temperature, will delineate the needed parameter range. Theoretically, using the low-alloy steel of Fig. 3 as an example, predictions of stresses for deformations up to about 0.5 per cent in 100,000 hr over the temperature range 900 to 1100 F should be obtainable within a few thousand hours test time.

Experience has shown that metallurgical instabilities tend to cast doubt on the validity of trading time for temperature in testing over such a wide range of variables. Consequently, extremely long-time tests are conducted at low stresses and actual service temperatures for adequate design precaution. While no actual comparisons are drawn here, it seems apparent that errors of a very small magnitude stem from extrapolation by Rabotnov's method, at least under several qualifying circumstances. In essence, a single tension curve and a single creep curve under proper testing conditions could serve to generate the entire isochronous family of creep curves at a given temperature. A more realistic approach might be the determination of three creep curves at various stresses and the given temperature so as to obtain the minimum number of isochronous curves necessary to evaluation. Under these conditions, the time required would be greater than that consumed in the parameter evaluation, but far less than that presently needed. In any event, the use of this method, at least as a complement to presently used techniques, should be considered.

TABLE V.—COMPARISON OF ACTUAL[a] AND ESTIMATED[b] VALUES OF $\phi(\epsilon)$ FOR LOW ALLOY STEEL.

Strain, per cent	$\phi(\epsilon)_{\text{tensile}}$	$\phi(\epsilon)^{b\,=\,0.33}_{\text{creep}}$	$\phi(\epsilon)^{b\,=\,0.30}_{\text{creep}}$
0.05	10 250	9 940	10 250
0.10	20 500	19 400	20 200
0.20	41 250	30 700	31 800
0.30	53 000	39 800	41 500

[a] Derived from tension curve.

[b] Derived from 10-hr and 1000-hr creep curves.

TABLE VI.—COMPARISON OF ESTIMATED AND ACTUAL CREEP STRESSES FOR LOW-ALLOY STEEL.[a]

Strain, per cent	Time, hr	Calculated Stress, psi	Actual Stress, psi	Error, per cent
0.05	30	8 200	8 300	−1.2
	100	7 550	7 700	−1.95
	300	6 850	6 900	−0.73
	3 000	5 120	5 100	+0.39
	10 000	4 200	3 800	+10.5
0.30	30	31 800	31 200	+1.6
	100	28 900	27 800	+3.95
	300	25 800	25 500	+1.18
	3 000	18 700	18 700	0
	10 000	15 200	14 300	+6.3

[a] Based on 10-hr and 1000-hr curves, $b = 0.30$.

DISCUSSION AND SUMMARY

The following answers to the questions originally proposed are presented:

1. Rabotnov's method can be used satisfactorily to correlate and extrapolate creep data from a wide class of materials. A least-squares analysis of the data shows that the maximum attainable accuracy of estimation by this method is highly dependent on the variables involved. The same analysis shows that a compromise leading to satisfactorily accurate estimations is practicable. Such compromise values appear to be those suggested by Rabotnov in his original work.

TABLE VII.—THE EFFECT OF SUBSTITUTING PLASTIC STRAIN FOR TOTAL STRAIN IN RABOTNOV'S EQUATION (LOW-ALLOY STEEL).[a]

Strain, per cent	Time, hr	Calculated Stress, psi	Actual Stress, psi	Error, per cent
0.05	30	24 500	21 900	+16.40
	100	19 600	19 200	+2.08
	300	15 300	15 800	−3.16
	3 000	8 400	8 400	0
	10 000	5 920	6 300	−6.03
0.10	300	22 600	21 000	+7.62
	3 000	13 200	12 800	+3.20
	10 000	9 540	8 750	+9.05
0.20	3 000	18 000	18 600	−3.22
	10 000	13 200	12 700	+3.94

[a] Based on tension curve and 1000-hr curve, $b = 0.33$.

2. In this study, materials with both stable and unstable structures were studied. The analysis shows that this factor is highly important. For plastic materials with highly stable structures, the correlation is excellent and the method highly insensitive to the change of correlating functions. On the other hand, for metallic materials where structure may change radically with time, the correlation over wide ranges of the variables does not seem to be as

good. In general, use of the value of 0.30 for b and computing both a and $\phi(\epsilon)$ from the data will lead to excellent correlations.

3. The use of the hot tension curve as a master curve in this method is open to question. As the least-squares analysis indicates, the best values of $\phi(\epsilon)$ do not correspond to values from the short-time tension curve. Using the alternate method of developing the master curve from the creep data leads to $\phi(\epsilon)$ values more nearly akin to those shown in the numerical analysis. Further, the use of these $\phi(\epsilon)$ values with $b = 0.30$ reduces the errors of estimation significantly. This may be connected to the stability of the alloy in the temperature range considered. In particular, the data for the 17-7 PH alloy indicate that when using the available tensile data, a value of $b = 0.15$ will give the best correlation. The resultant errors are large. However, when predicting the master curve without reference to tensile data, the value of $b = 0.30$ is about optimum. The results would tend to show that using the tension curve at increasingly large strains leads to increasingly large errors.

4. The design potential of this method should be good. Presenting the data in a manner that allows a direct approach to the question of material strength for a given deformation in a specified time period is an obvious advantage. Aside from this, proper use of the method appears to sustain very small errors in both interpolation and extrapolation of data.

Acknowledgment:

The author's attention was first drawn to this subject by B. M. Wundt of the Large Steam Turbine-Generator Dept., General Electric Co. He was highly instrumental in encouraging this effort.

The author also wishes to acknowledge the help of E. J. Tracey of the Materials and Processes Laboratory for preparing the visual material shown in this paper, and also of members of the Mathematical Analysis Unit of General Engineering Laboratory, General Electric Co., for their aid in the numerical analysis programming.

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(1) Y. N. Rabotnov, "Some Problems on the Theory of Creep," Vestnik Moskovskovo Universiteta, No. 10, pp. 81–91 (1948), (Translated and available as Natl. Advisory Comm. Aeronautics *Technical Memorandum 1353*).

(2) S. S. Manson and W. F. Brown, "A Survey of the Effects of Nonsteady Load and Temperature Conditions on the Creep of Metals," *Proceedings*, Fourth Sagamore Conference, August, 1957 (Syracuse University Research Inst. Met. pp. 497–582).

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DETERMINATION OF COMPRESSIVE, BEARING, AND SHEAR CREEP OF SHEET METALS*

By J. R. Kattus[1] and H. L. Lessley[1]

Synopsis

A description is given of facilities to determine the compressive, bearing, and shear creep properties of sheet metals up to about 1200 F. The compressive and bearing determinations are carried out in loading fixtures that contain cartridge heaters as integral parts. These heating units, which obviate the need for conventional furnaces, provide extremely good temperature uniformity. Accurate strain measurements are facilitated because the extensometer can be positioned quite close to the specimen, thus eliminating mechanical errors that occur in the long extension arms required with furnace heating. Shear creep-rupture determinations are carried out on both sheet-type and pin-type specimens in conventional creep furnaces. Examples are given of the results obtained on titanium alloys.

During recent years, a tremendous amount of information has been obtained and published on the creep characteristics of metals under tensile loads. Data of this type are required for the selection of alloys and for the design of structures that must sustain loads at elevated temperatures. For many elevated-temperature applications, however, compressive, shear, or bearing loads are more prevalent than tensile loads. Only limited research has been done and limited data are available on the creep characteristics of metals when they are subjected to such loads. When such data are needed, it is frequently assumed that tensile creep properties, with the application of a questionable conversion factor, can be used to predict creep characteristics under the other loading conditions. Such assumptions have not been proved to be consistently accurate or reliable. For optimum reliability and economy in materials selection and in structural design, investigations of the creep characteristics of metals under compressive, shear, and bearing loading are clearly needed.

The purpose of this paper is to describe facilities that have been developed at Southern Research Inst. to determine the creep and creep-rupture properties of sheet metals under constant compression, bearing, and shear loads. These facilities have been used primarily in evaluations of titanium alloys at temperatures up to 1000 F for creep times up to 500 hr. They should be useful for all types of metals at temperatures up to 1200 F, and for unlimited times.

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* Presented at the Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] Head of Metallurgy Division and Associate Metallurgist, respectively, Southern Research Inst., Birmingham, Ala.

FACILITIES

All evaluations are carried out with dead-weight loading applied through lever systems. Commercial creep machines are used for bearing and shear loading, whereas special loading frames designed and built at Southern Research Inst. are used for compression loading. In the bearing- and compression-creep evaluations, strain-time curves are recorded, but only rupture time is determined in the shear-creep evaluations. Two types of shear specimens are used: pin-type or double shear and sheet-type or single shear.

In all of the evaluations, one thermocouple is used in conjunction with a controller and suitable proportioning equipment to provide temperature control within ±3 F of the desired temperature. Two or three others are used for precise monitoring of temperature level and uniformity with a portable potentiometer.

Compression:

The creep frame, subpress, fixture, and recorder used in the compression-creep determinations are shown in Fig. 1. Figure 2 shows a cross-section of the fixture, and Fig. 3 shows the disassembled fixture. Buckling in the compression sheet specimens, which are $\frac{5}{8}$ in. wide by $2\frac{5}{8}$ in. long, is prevented by nickel support blocks, nickel being used because of its excellent oxidation resistance. To facilitate temperature uniformity, the supporting faces of these blocks are flush. They are polished and lubricated with molybdenum disulfide to minimize friction. Eight 85-w cartridge heaters, which obviate the need for a furnace, are inserted into the nickel support blocks. With proper spacing of these heaters, temperature uniformity of ±3 F over the 2-in. gage length is achieved at temperatures up to at least 1000 F. The degree of support, which is controlled by two adjustable band clamps around the outside of the fixture, is measured in terms of the load required to cause free specimen slippage prior to the application of the compression load. For large compression loads exceeding the yield strength, free-slippage forces as high as 150 lb have been necessary to restrict specimen buckling, whereas free-slippage forces as low as 5 lb have been sufficient with relatively low loads. It is believed that this wide range of specimen support has little effect on the deformation characteristics of the specimens because compressive modulus-of-elasticity values determined up to this maximum degree of support at various temperatures have been consistently within ±5 per cent of the tensile-modulus values, a normal degree of variation.

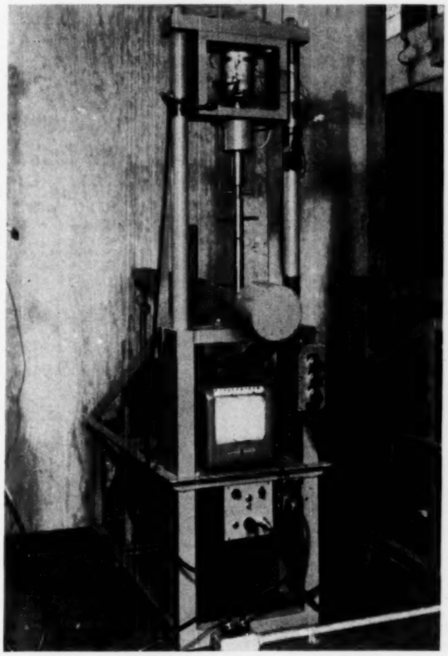

FIG. 1.—Equipment for Compression Creep.

The compact design of the compression fixture allows the use of a compact extensometer with the transducer mounted only 3 to 4 in. from the specimen. This type of extensometer design prevents possible mechanical errors associated with the long extension arms that are needed to hold the transducers out of the heated zone when normal furnace heating is used. The compression extensometer is illustrated in Fig. 4. The contact points fit into light punch marks on opposite edges of the specimen at the gage points, the horizontal arms transmitting the deformation between the gage points to the core and coil of two differential transformers. Taut nylon monofilaments that support the cores are guided through eyelets at each end of the coils to maintain concentricity between the cores and the coils. The two halves of the extensometer are positioned laterally on a horizontal dovetail slide that is supported by the compression fixture. Figure 5 shows the extensometer in place.

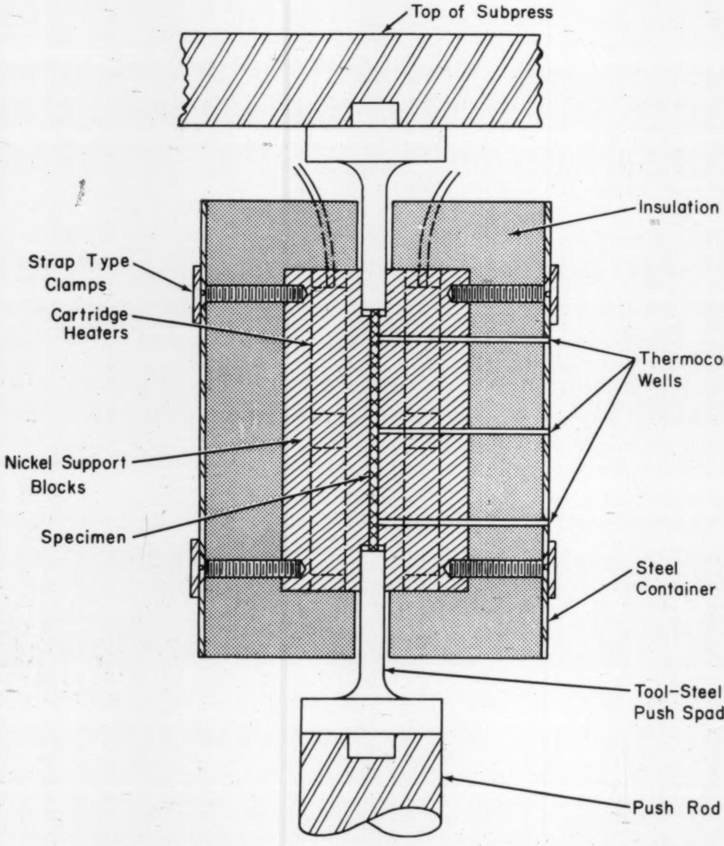
Top of Subpress
Insulation
Strap Type Clamps
Cartridge Heaters
Thermocouple Wells
Nickel Support Blocks
Specimen
Steel Container
Tool-Steel Push Spade
Push Rod

Fig. 2.—Cross-Section of Compression Fixture Facing Edge of Specimen.

Excitation for the differential transformers in the extensometer is provided by a stable source of voltage at 15,000 cps. The combined output of the differential transformers is rectified by a phase-sensitive detector and attenuated by a voltage divider. This signal, which is calibrated in terms of specimen deformation, is fed into a strip-chart recorder that provides a curve of creep deformation as a function of time.

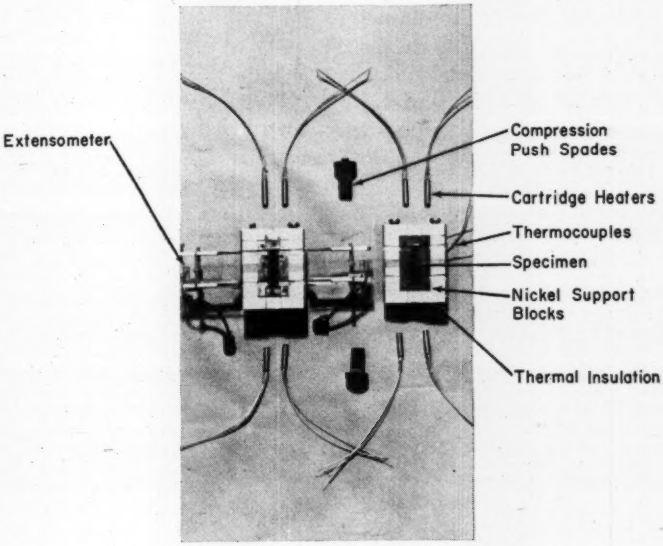
Extensometer
Compression Push Spades
Cartridge Heaters
Thermocouples
Specimen
Nickel Support Blocks
Thermal Insulation

FIG. 3.—Disassembled Compression Fixture.

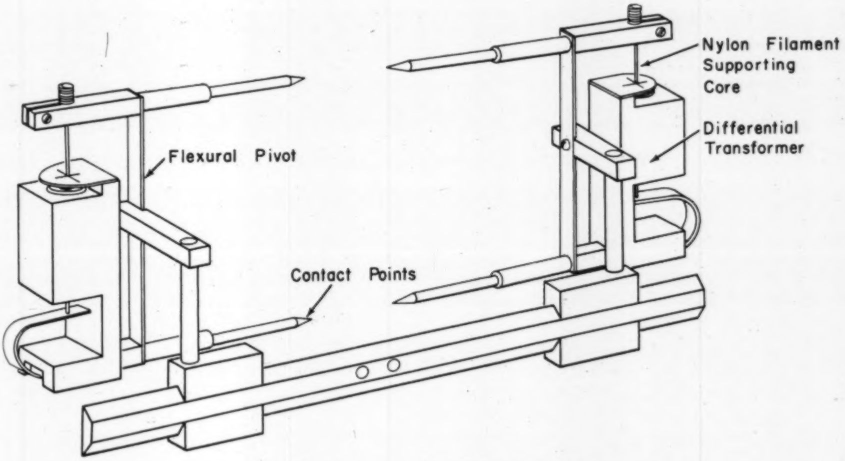
Nylon Filament Supporting Core
Differential Transformer
Flexural Pivot
Contact Points

FIG. 4.—Compression Extensometer.

Bearing:

The bearing heating and loading fixtures and extensometers, which are shown mounted in a creep machine in Fig. 6, are also compact in construction. Heat for the specimens is provided by a pair of 200-w electrical disk heaters in each fixture as illustrated by the cross-sectional view in Fig. 7. Figure 8 shows a disassembled bearing fixture.

The bearing specimens, which are flat sections of sheet with over-all dimensions of $1\frac{1}{2}$ by $4\frac{3}{4}$ in., are clevis-gripped at the lower end through a $\frac{1}{2}$-in. diameter hole and are loaded at the top end through a $\frac{5}{16}$-in. diameter bearing hole with a ratio of edge distance to diameter of 2. With copper heat-diffusion disks placed between the specimen and the heaters, temperature uniformity of ±3 F is maintained in the half of the specimen that contains the bearing hole.

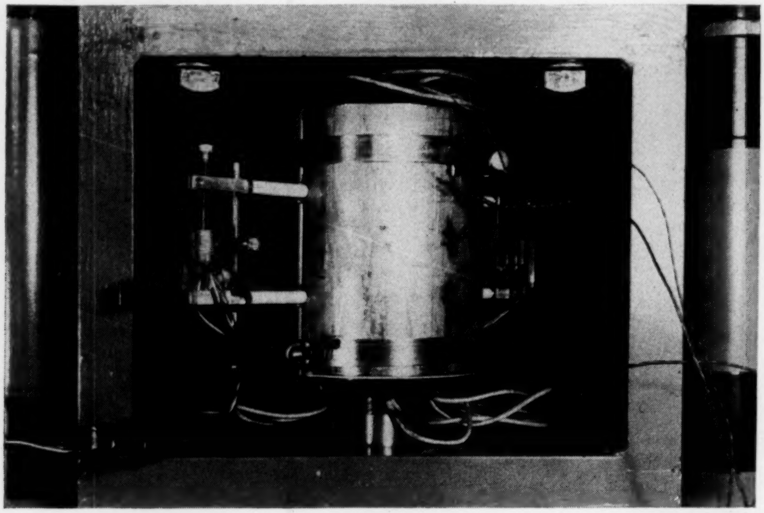

FIG. 5.—Compression Fixture, Extensometer, and Subpress.

The extensometer and system for recording time-deformation curves are exactly the same as those used for compression creep except, as shown in Fig. 6, the upper pair of extensometer arms is eliminated and the dovetail slide, which supports the extensometer, is mounted on the loading clevis. The lower pair of arms is attached through point contacts to opposite edges of the specimen on a line tangent to the loaded side of the bearing hole. With this system, the deformation in the bearing hole is detected by the two differential transformers, the combined output being calibrated in terms of either total deformation or per cent of bearing-hole diameter.

Shear:

Both the single- and double-shear specimens are heated with temperature uniformity of ±3 F in conventional creep furnaces. Figure 9(*a*) shows a double-shear specimen and a disassembled loading fixture. When the specimen is assembled with the fixture and a load is applied, shear stresses are developed across the two cross-sections of the specimen that are in the same planes as the interfaces between the two halves of the pull rods. Shear creep-rupture occurs in these two cross-sections.

The single-shear specimens contain two offset slots at 45 deg to the major axis as shown in Fig. 9(*b*). When the specimen is loaded in tension as shown in the diagram, shear stresses are developed in the $\frac{3}{16}$-in. section of the major axis between the ends of the slots. Shear-type creep rupture has occurred in this section in the vast majority of specimens that have been evaluated, which includes a variety of high-strength titanium and steel alloys. Only in a high-strength titanium alloy with tensile elongation of less than 1 per cent did tensile type failures occur, the rupture propagating from the base of a slot to the opposite edge of the specimen. Generally, the single-shear specimen shown in Fig. 9(*b*) is suitable except for specimens with extremely low ductility and corresponding high notch sensitivity.

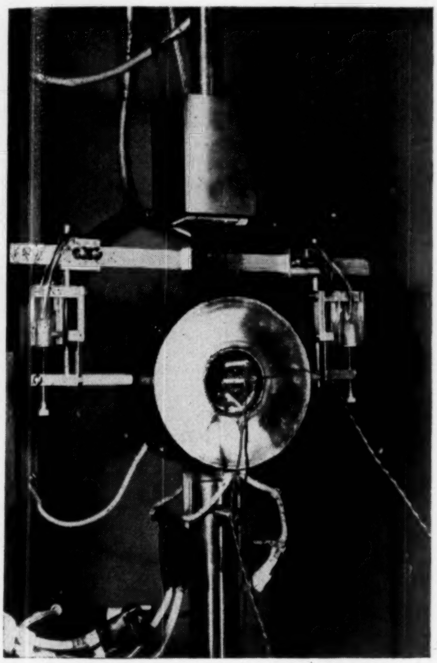

FIG. 6.—Bearing Fixture and Extensometer in Creep Machine.

TEST RESULTS

Design-type creep curves for various types of loading are illustrated in Figs. 10 and 11 for a titanium alloy sheet (Ti-2.5Al-16V solution treated and aged). In these curves, rupture times or times to certain percentages of creep strain are shown as functions of nominal stress at constant temperatures of 600, 700, and 800 F. For comparative purposes, the short-time tensile properties of this alloy are given in Table I.

Figure 10(*a*) illustrates design curves for 0.1 per cent and 0.5 per cent compressive creep. At 700 and 800 F the data are reasonably consistent showing a normal amount of scatter. At 600 F, fewer data points are available because, at the high loads required to cause appreciable creep, a considerable number of specimens buckled in spite of large supporting pressures, measured in terms of free-slippage forces of 150 lb.

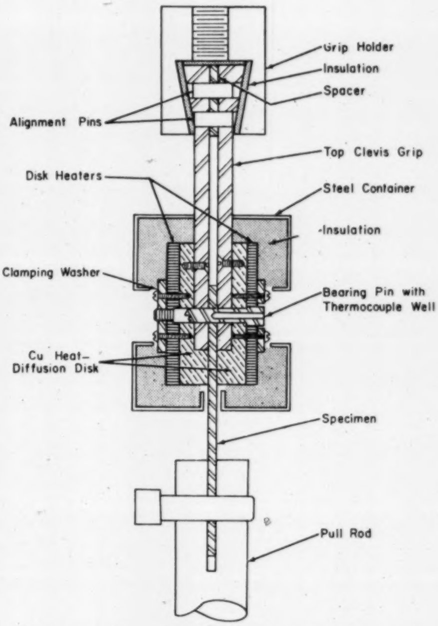

FIG. 7.—Cross-Section of Bearing Fixture Facing Edge of Specimen.

Corresponding tensile creep data obtained on the same material[2] by the Georgia Division, Lockheed Aircraft Corp., are shown in Fig. 10(*b*). Comparing Figs. 10(*a*) and 10(*b*), it will be seen that the compressive creep strength is consistently higher than the tensile

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[2] P. J. Hughes, W. M. McGee, and O. L. White, "The Determination of Mechanical Property Design Data for Heat Treated Titanium Alloys Being Produced by the DOD Sheet Rolling Program," Contract No. AF 33(616)-6346, *Progress Report No. ER-3030, Vol. VII*, Lockheed Aircraft Corp., Marietta, Ga., Dec. 1960.

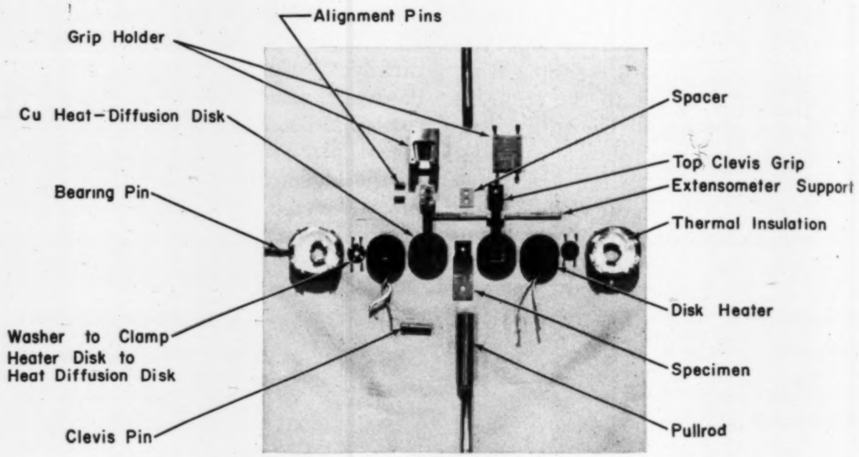
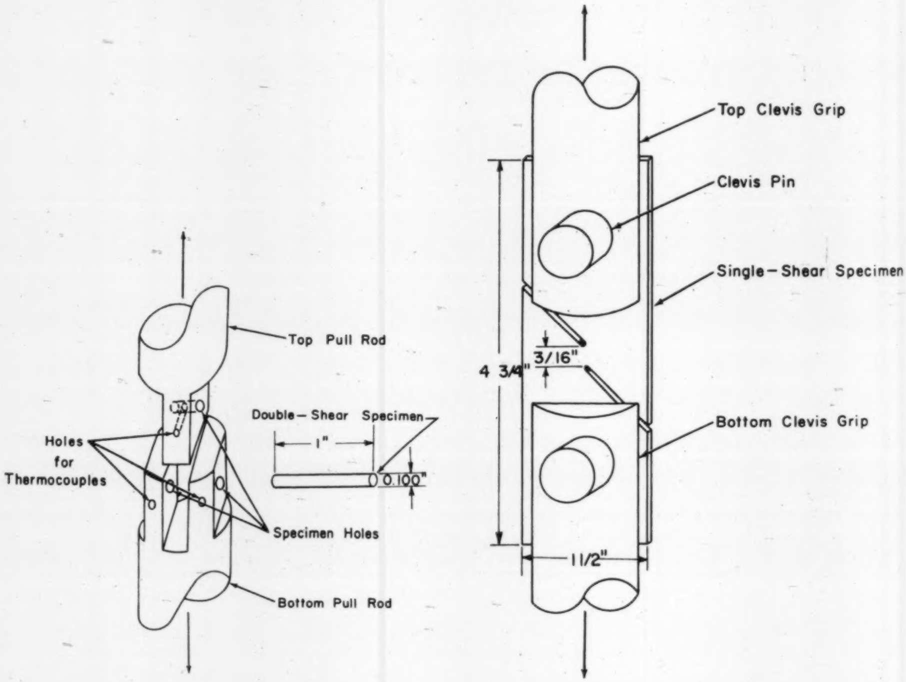
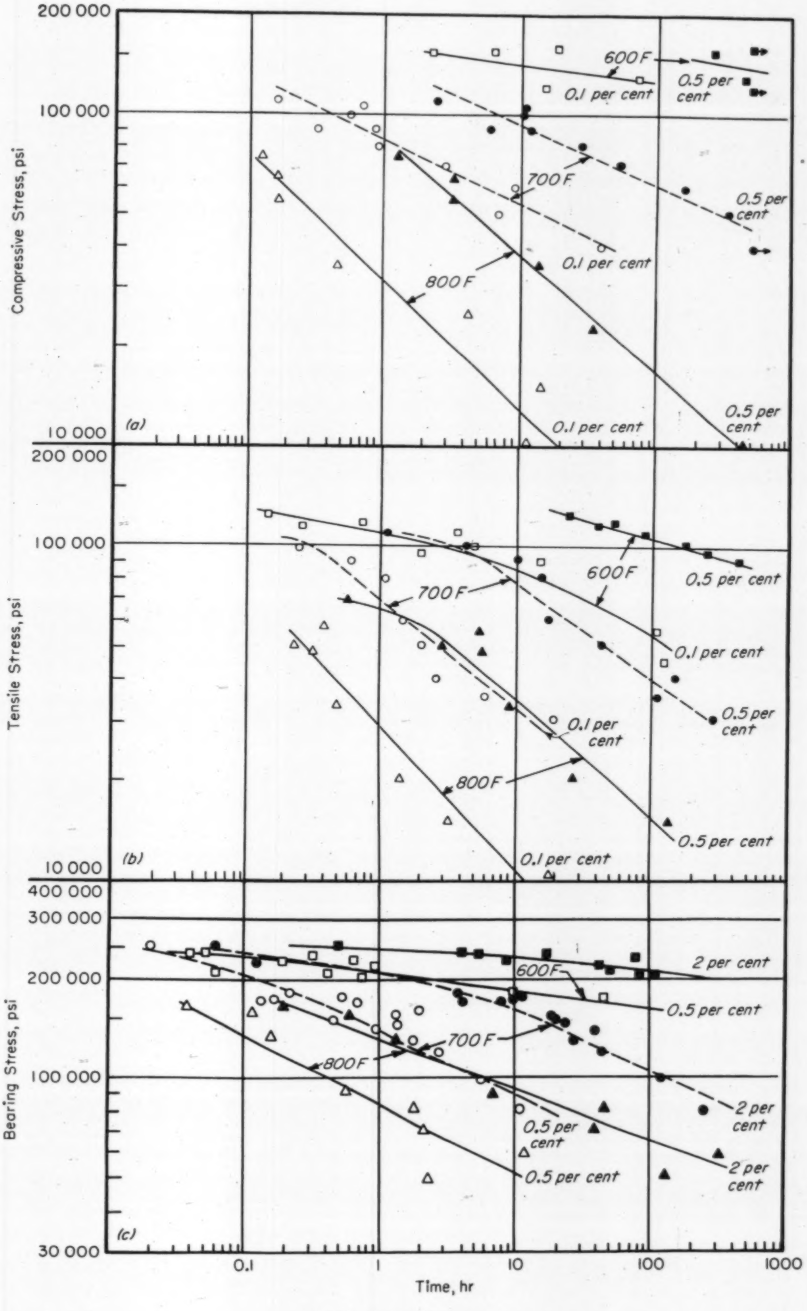

Alignment Pins
Grip Holder
Cu Heat-Diffusion Disk
Bearing Pin
Washer to Clamp Heater Disk to Heat Diffusion Disk
Clevis Pin
Spacer
Top Clevis Grip
Extensometer Support
Thermal Insulation
Disk Heater
Specimen
Pullrod

FIG. 8.—Disassembled Bearing Fixture.


Top Pull Rod
Holes for Thermocouples
Double-Shear Specimen
1"
0.100"
Specimen Holes
Bottom Pull Rod
Top Clevis Grip
Clevis Pin
Single-Shear Specimen
4 3/4"
3/16"
Bottom Clevis Grip
11/2"

(*a*) Double-shear specimen and fixture. (*b*) Single-shear specimen and fixture.

FIG. 9.—Shear Specimens and Loading Fixtures.



(*a*) Compressive creep.
(*b*) Tensile creep.
(*c*) Bearing creep.

FIG. 10.—Creep Design Curves for Ti-2.5Al-16V Alloy Sheet, Solution Treated and Aged.

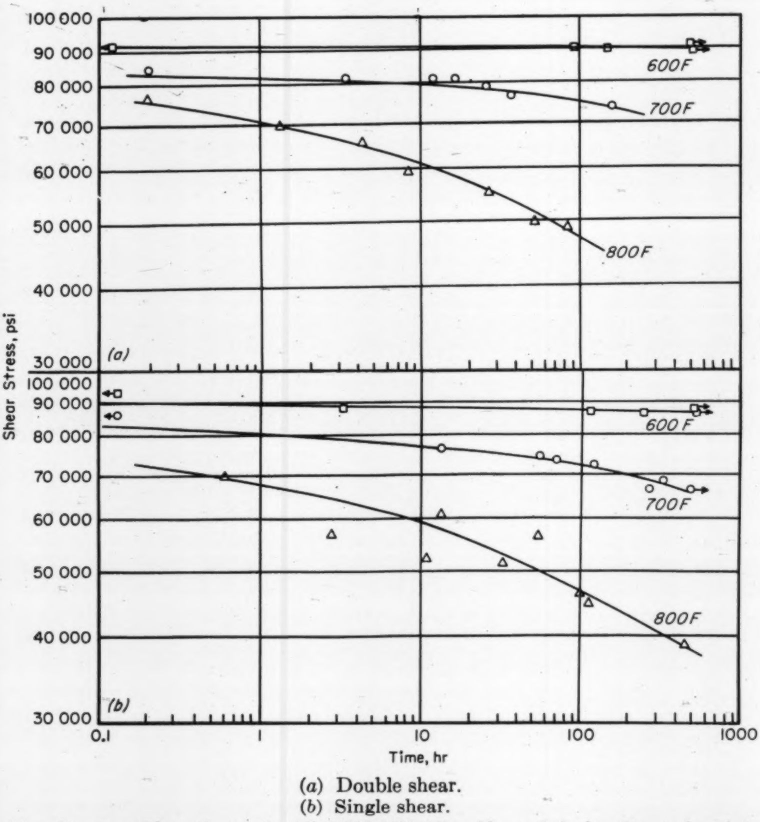
creep strength, the differences being quite significant at 600 and 700 F, but negligible at 800 F. Upon first consideration, it might be reasoned that the differences at the lower temperatures are a result of faulty experimental techniques, namely, the large supporting pressures applied to the compression specimens. Although this possibility is recognized and is being investigated, the following facts indicate that the differences are real and that the experimental techniques are satisfactory:

1. Generally, the amount of strain that occurred in compression-creep specimens during the application of load approached that in tension-creep specimens.

2. The supporting pressures were the same at 700 and 800 F, being sufficient to require a force of 50 lb to cause free specimen slippage. Only at 600 F was a larger supporting pressure—one sufficient to require a force of 150 lb to cause free specimen slippage—needed to minimize the occurrence of buckling.

TABLE I.—TENSILE STRENGTH OF Ti-2.5Al-16V ALLOY SHEET AFTER SOLUTION TREATMENT AND AGING.

Temperature, deg Fahr	Ultimate Tensile Strength, psi	Yield Strength, 0.2 per cent Offset, psi	Elongation in 2 in., per cent
80	174 000	158 000	6.2
600	149 000	128 000	5.9
800	137 000	108 000	7.5



(*a*) Double shear.
(*b*) Single shear.

FIG. 11.—Rupture-Time Curves for Ti-2.5Al-16V Alloy Sheet, Solution Treated and Aged.

5. Accurate elastic-modulus values were obtained in compression with both of these levels of specimen support.

Design-type curves for 0.5 per cent and 2 per cent bearing creep strain are illustrated in Fig. 10(*c*). The two degrees of creep strain are in terms of percentages of the bearing-hole diameter; bearing stress is defined as the load divided by the product of the specimen thickness and bearing-hole diameter. The scatter in the bearing-creep data is quite small at 600 F but increases somewhat with increasing temperatures.

Rupture times as functions of shear stress are shown in Figs. 11(*a*) and (*b*), Fig. 11(*a*) pertaining to double-shear specimens and Fig. 11(*b*) to single-shear specimens. The double-shear and single-shear rupture strengths are almost identical.

SUMMARY

The data presented in this paper represent some of the initial results obtained on recently developed facilities for the determination of the compressive, bearing, and shear creep properties of sheet metals. Although the data and procedures are believed to be valid, it must be recognized that a need for verification and possible improvements exist, particularly with regard to the compression-creep determinations. It is hoped that this paper will stimulate discussion and reporting of additional information on the subject from other sources. Such pooling of information should lead to the eventual development of standard procedures.

Acknowledgments:

Much of the design and development of the facilities was done by C. R Bieber, J. B. Preston, and O. V. Rogers. Mr. Bieber and W. L. Rogers carried out most of the creep determinations.

All of the work was conducted under the sponsorship of the Wright Air Development Division, Air Research and Development Command. The prime contract, AF 33(616)-6346, was vested in the Lockheed Aircraft Corp., Marietta, Ga., and the work at Southern Research Inst. was conducted under subcontract J4-21404.

DISCUSSION

MR. E. E. REYNOLDS.[1]—How do these data compare with tension creep data?

MR. J. R. KATTUS (*author*).—The only comparisons we have made so far are between some compression and tension creep data; we have not tried to correlate any shear or bearing data with tension creep data. As I pointed out, at 600 and 700 F the Ti-2.5Al-16V had higher creep strength in compression than in tension, but at 800 F they were practically the same.

MR. EDWARD L. HORNE.[2]—I would like to ask two questions: Were the effects of friction on the compression test results and the magnitude of support involved taken into account? Are there comparative data with other organizations using somewhat different techniques and equipment?

MR. KATTUS.—With regard to friction, some requirements have been set up for the DOD titanium sheet rolling program for qualifying compression fixtures. The method of qualifying requires running some short-time stress-strain curves in the fixtures and determining the modulus of elasticity for some materials for which the modulus in tension is known. If the compression modulus determined with this equipment is within 5 per cent of that determined by accurate tensile means, the equipment qualifies.

We have not made many comparisons yet between the results we obtained and those which other people have obtained. This will be done to some extent; comparisons are to be made between our compression creep data and Lockheed's tensile creep data that they are to run in the future.

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[1] Chief Research Metallurgist, Allegheny Ludlum Steel Corp., Brackenridge, Pa.

[2] Supervisory Materials Engineer, Aeronautical Systems Division, Materials Central, Wright-Patterson Air Force Base, Ohio.

DETERMINATION OF CREEP OF STRUCTURAL JOINTS FROM SIMPLE TENSILE CREEP*

By Joseph Marin[1]

Synopsis

This paper outlines an approximate method for the theoretical determination of the creep deformation in structural joints based upon simple tensile creep stress-strain-time relations for the material. It should be noted that the problem is a complex one and that it was necessary to make a number of assumptions in order to develop a usable theory. In addition to providing a prediction of the possible creep deformation that may occur in a riveted joint, the analysis given below can be used to guide the designer in the selection of the best material. In other words, a comparison of the theoretically determined deformations of structural joints using various materials gives a better basis for comparison of performance than a comparison of the simple tensile creep deformations. In the analysis of the creep deformation of riveted or bolted joints, a theory was also developed for predicting the transverse shear creep deformations from simple tensile creep.

A review of publications on creep of riveted joints (**4–6**)[2] shows that, except for theories developed by the author in references (**1**), (**2**), and (**3**), the available information is primarily confined to experimental studies. If evaluations on creep of riveted joints are based on tests alone, then a new test program must be conducted for each new riveted joint configuration and each new material. It would, of course, save considerable time and effort if the creep deformation of all riveted joints could be theoretically predicted from the basic simple tensile-creep data of the material. This paper attempts to provide such a prediction for a single-riveted joint of the butt type.

Creep of Single-Riveted Butt Joint

Figure 1 shows a single-riveted butt joint subjected to an axial force *F*. To define the creep deformation between two points *A* and *G* of this joint, it is first necessary to note that the total creep deformation is made up of the following creep deformations:

1. Deformation e_1 for the length L_1 of the plate,
2. Deformation e_2 for the length L_2 of the plate,
3. Deformation e_3 for the length L_3 of the plate,
4. Deflection e_R of the rivet,
5. Deformation e_4 for the length L_4 of the plate,
6. Deformation e_5 for the length L_5 of the plate, and

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* Presented at the Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] Department of Engineering Mechanics, The Pennsylvania State University, University Park, Pa.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

7. Deformation e_6 for the length L_6 of the plate.

The lengths L_1 and L_6 correspond to lengths for which the creep deformation is essentially uniform across the plate. The locations of these lengths of constant deformation would have to be determined by experiment. If this is not possible, an estimate of the approximate value of these lengths could be made. Errors produced by such estimates should not greatly influence the final results. The total deformation for the riveted or bolted joint between the points A and G is the sum of all the component parts of the creep deformation or

$$e = e_1 + e_2 + e_3 + e_R + e_4 + e_5 + e_6 \quad (1)$$

The e values on the right-hand side of Eq 1 can now be determined. To find e_1 and e_6 it will be assumed that those deformations are the same as those produced in a tension bar without a riveted joint. Then the values of e_1 and e_6 equal the unit strain multiplied by the lengths L_1 and L_6. The unit tensile-creep strain will be assumed to be expressed by Eq 1. That is,

$$\epsilon = kS^n \quad (2)$$

where S = the stress and k is the time function expressed by

$$k = k_1 + k_2(1 - e^{-qT}) + BT \quad (3)$$

where:

k_1, k_2, n, B and q = tensile creep constants for a given temperature,

e = the base of natural logarithms, and

T = the time.

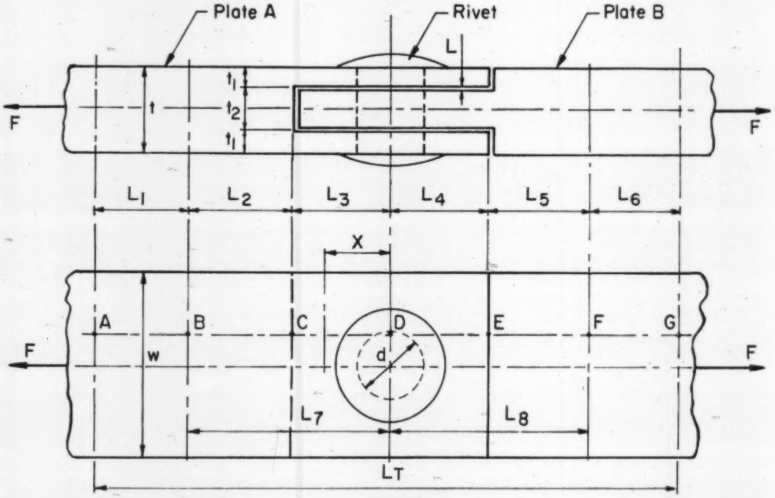


FIG. 1.—Single-Riveted Butt Joint.

Then the values of e_1 and e_6 become

$$e_1 = \epsilon L_1 = kS^n L_1 = k\left(\frac{F}{wt}\right)^n L_1 \quad (4)$$

$$e_6 = \epsilon L_6 = kS^n L_6 = k\left(\frac{F}{wt}\right)^n L_6 \quad (5)$$

where:

F = the axial load,

w = the width of the member (Fig. 1), and

t = the thickness of the member (Fig. 1).

To determine the deformation e_3 for the length CD, let the tensile stress at point D in the plate be S_D and the stress at B be S_B'. If S_B is the stress that would have existed at B for a plate of the same thickness, t, at B and D, then it can be assumed that

$$S_B = \left(\frac{t}{2t_1}\right)(S_B') \quad \ldots\ldots\ldots\ldots (a)$$

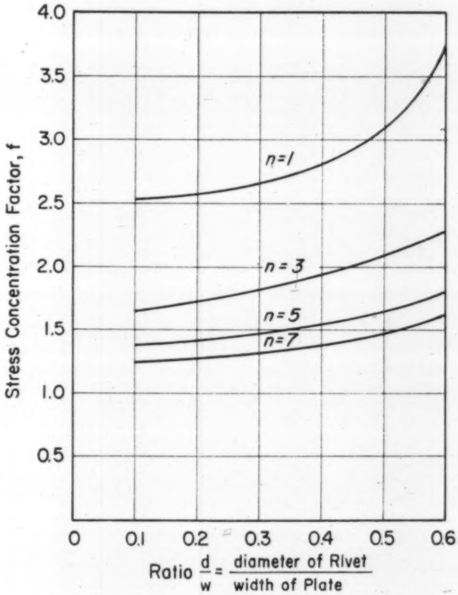


FIG. 2.—Stress Concentration Factor, f.

An approximate value of the stress S_D at the edge of the rivet hole at D was evaluated by the author in reference (1). To determine the creep deformation between B and D, some assumption will have to be made regarding the manner in which the stress varies between B and D. A reasonable approximation is to assume that the stress variation between B and D is linear, where the stress at B is the stress for a plate of thickness $2t_1$. That is,

$$\frac{S_D - S_B}{S_x - S_B} = \frac{L_7}{L_7 - x}$$

or

$$S_x = S_D\left(1 - \frac{x}{L_7}\right) + S_B\left(\frac{x}{L_7}\right) \quad \ldots (b)$$

Using Eq 2, the total creep deformation for the length CD becomes

$$e_3 = \int_0^{L_3} \epsilon_x \, dx = \int_0^{L_3} (kS_x^n) \, dx \quad \ldots (c)$$

Placing values of the stress from Eq b in Eq c and integrating the resulting equation,

$$e_3 = \frac{L_7 k}{(S_B - S_D)(n+1)}$$

$$\cdot\left[\left(\frac{L_2}{L_7} S_D + \frac{L_3}{L_7} S_B\right)^{n+1} - S_D^{n+1}\right] \quad (d)$$

If f is the stress concentration factor at the edge of the hole at D, then

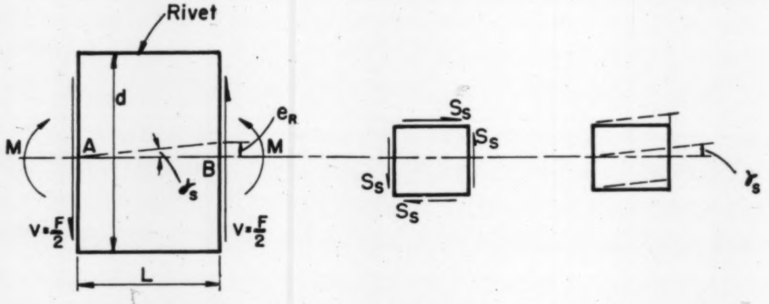
$$f = \frac{S_D}{S_B}, \quad \text{or} \quad S_D = fS_B = \left(\frac{ft}{2t_1}\right) S_B'$$

$$= \left(\frac{ft}{2t_1}\right)\left(\frac{F}{wt}\right) = \left(\frac{fF}{2t_1 w}\right) \quad (6)$$

Placing the value of S_D from Eq 6 in Eq d,

$$e_3 = \frac{kL_7[F/(2wt_1)]^n}{(n+1)(f-1)} \cdot \left[f^{n+1} - \left(f\frac{L_2}{L_7} + \frac{L_3}{L_7}\right)^{n+1}\right] \quad (7)$$

Equation 7 defines the creep deformation e_3 in terms of the dimensions L_2, L_3, t, t_1, n; the material and time constants k and n; the creep stress concentration factor f; and the load F. The material-time factor k as expressed by Eq 3 can be evaluated from tensile creep data for a given time T. Values of the stress concentration factors f, as obtained in reference (1), are plotted in Fig. 2 for various values of n.



(*a*) Free body diagram of rivet. (*b*) Stresses along *AB*. (*c*) Strains along *AB*.

FIG. 3.—Shear Deformation of a Rivet.

The creep deformation e_2 for the length *BC* is given by

$$e_2 = \int_{L_3}^{L_7} \epsilon_x \, dx = \int_{L_3}^{L_7} k(S_x')^n \, dx \quad \ldots(e)$$

Using Eq *b*,

$$S_x' = \left(\frac{2t_1}{t}\right) S_x$$

$$= \left(\frac{2t_1}{t}\right) \cdot \left[\left(1 - \frac{x}{L_7}\right)(S_D) + \frac{x}{L_7} S_B\right] \quad ..(f)$$

Placing the value of the stress from Eq *f* in Eq *e* and integrating the resulting equation,

$$e_2 = \frac{kL_7[F/(wt)]^n}{(n+1)(f-1)} \cdot \left[\left(f\frac{L_2}{L_7} + \frac{L_3}{L_7}\right)^{n+1} - 1\right] \quad ..(8)$$

Equation 8 defines the creep deformation e_2 in terms of the dimensions L_2, L_3, $L_7 = L_2 + L_3$, t, t_1, w; the material and time constants k and n; the creep stress concentration factor f; and the load F.

The creep deformation e_4 can be obtained from Eq 7 provided $2t_1$ is replaced by t_2, L_3 is replaced by L_4, L_2 is replaced by L_5, and L_7 is replaced by L_8. Then

$$e_4 = \frac{kL_8[F/(wt_2)]^n}{(n+1)(f-1)} \cdot \left[f^{n+1} - \left(f\frac{L_5}{L_8} + \frac{L_4}{L_8}\right)^{n+1}\right] \quad ..(9)$$

In the same way for the deformation e_5, Eq 8 can be used provided L_3 is replaced by L_4, L_2 by L_5, and L_7 by L_8. Then

$$e_5 = \frac{kL_8[F/(wt)]^n}{(n+1)(f-1)} \cdot \left[\left(f\frac{L_5}{L_8} + \frac{L_4}{L_8}\right)^{n+1} - 1\right] \quad (10)$$

An analysis of the creep deformation in the longitudinal direction of the joint produced by the deformation of the rivet is difficult to determine, because the effective length of the rivet producing longitudinal deformation is not known. In view of this difficulty, the effective rivet length, L, can be selected as the clearance between plates or some greater value which provides for possible clearance between the rivet and rivet hole. In any case, it can be assumed that the rivet is subjected to forces as shown in Fig. 3(a). Since the length L is small compared to the diameter d of the rivet, it will be assumed that the deformation e_R of the rivet is due to shear only and that the deformation due to bending can be neglected. Then the deformation e_R is

$$e_R = \gamma_s L \quad (g)$$

It can be shown that at the centroidal axis AB the shear strain γ_s (Fig. 3(a)) in terms of the shear stress S_s is

$$\gamma_s = 3^{\frac{n+1}{2}} kS_s^n \quad (h)$$

where k and n are the tensile creep constants in Eq 2 (3).

Reference (3) also shows that the shear stress S_s at the centroidal axis is

$$S_s = \beta\frac{V}{A} = \beta\left(\frac{F}{2A}\right) \quad (i)$$

where $A = \pi d^2/4$ = the cross-sectional area of the rivet, and the approximate value of β is

$$\beta = \frac{\pi(6n+1)(4n+1)(2n+1)}{4(5n+1)(3n+1)(n+1)} \quad (j)$$

Then from Eqs (g), (h), (i), and (j) the rivet deformation e_R becomes

$$e_R = 3^{\frac{n+1}{2}} kL\left(\frac{2F}{d^2}\right)^n \cdot \left[\frac{(6n+1)(4n+1)(2n+1)}{4(5n+1)(3n+1)(n+1)}\right]^n \quad (11)$$

With the values of e_1, e_2, e_3, e_R, e_4, e_5, and e_6 defined by Eqs 4, 5, 7, 8, 9, 10 and 11, Eq 1 gives the total deformation. An outline of the steps involved in the determination of the total creep deformation is summarized below.

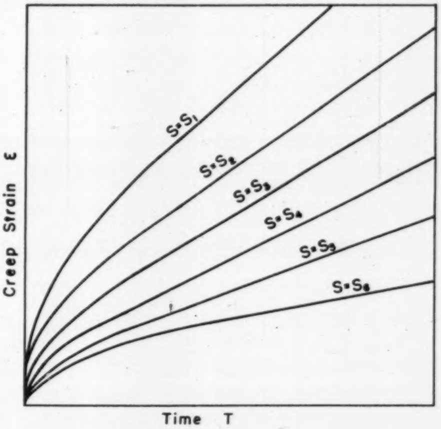


FIG. 4.—Tensile Creep-Time Stress Curves.

Procedure for Calculation of Total Creep Deformation:

1. From tensile creep-time test results (Fig. 4) the values of the creep tension constants k_1, k_2, q, B, and n, as expressed in Eqs 2 and 3, can be determined.
2. Values of dimensions L_1, L_2, L_3, L_4, L_5, L_6, L, t, t_1, t_2, d, and w for the riveted butt joint are selected for the given application.
3. Using Eqs 4 and 5, e_1 and e_6 are determined for a known k value based on a time T.
4. From Fig. 2, the stress concentra-

tion factor f for the known values of d/w and n is obtained.

5. Using Eqs 7, 8, 9, and 10, the values of e_2, e_3, e_4, and e_5 can be calculated since k, n, f and the dimensions of the riveted joint are known.

6. The deflection of the rivet can be calculated using Eq 11.

7. With values of e_1 to e_6 and e_R known, Eq 1 gives the total creep deformation of the riveted butt joint.

Example.—Compare the total creep deformation of a tension member subjected to an axial load F of width w, thickness t, and length L_T with that of a riveted butt joint of length L_T as shown in Fig. 1. Given: $n = 3$, $t_2 = 2t_1 = t/2, t/d = 2, w/t = 2, L/d = 1/9$, $L_1 = L_2 = L_3 = L_4 = L_5 = L_6 = L_T/6$, $w = L_T/3$ and $\epsilon = kS^n$.

The creep deformation of a tension member of width w, length L_T, thickness t, and without a rivet is

$$e_T = \epsilon L_T = kS^nL_T = k\left(\frac{F}{wt}\right)^n L_T \quad (12)$$

From Eqs 1 and 12, the ratio of the total deformation of the riveted to the nonriveted member is

$$\frac{e}{e_T} = (e_1 + e_2 + e_3 + e_R + e_4 + e_5 + e_6)/e_T$$

or

$$\frac{e}{e_T} = 2\left(\frac{e_1}{e_T} + \frac{e_2}{e_T} + \frac{e_3}{e_T} + \frac{e_R}{2e_T}\right) \quad (13)$$

Placing values of the deformations from Eqs 4 to 11 in Eq 13:

$$\begin{aligned}\frac{e}{e_T} = {} & 2\left(\frac{L_1}{L_T}\right) \\ & + \left(\frac{2L_7}{L_T}\right)\frac{\left[\left(f\frac{L_2}{L_7} + \frac{L_3}{L_7}\right)^{n+1} - 1\right]}{(n+1)(f-1)} \\ & + \left(\frac{2^{n+1}L_7}{L_T}\right)\frac{\left[f^{n+1} - \left(f\frac{L_2}{L_7} + \frac{L_3}{L_7}\right)^{n+1}\right]}{(n+1)(f-1)} \\ & + 3^{\frac{n+1}{2}}\left(\frac{L}{L_T}\right)\left(\frac{2wt}{d^2}\right)^n \\ & \left[\frac{(6n+1)(4n+1)(2n+1)}{4(5n+1)(3n+1)(n+1)}\right]^n \quad (14)\end{aligned}$$

From Fig. 2, since $\frac{d}{w} = 0.25$ and $n = 3$, $f = 1.70$:

$$\frac{e}{e_T} = 0.33 + 0.55 + 9.57 + 105.17 = 115.62$$

Equations similar to the above can be written for single-riveted lap-type joints.

SUMMARY

This paper provides an approximate method for predicting the creep deformation of a riveted joint from the creep constants of the material in simple tension. It should be noted that although test results may show that the theoretical relations obtained give only approximate deformations, the theory does provide a measure of the factors which influence the creep deformation of a riveted joint.

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(2) Joseph Marin, "Determination of the Creep Deflection of a Rivet in Double Shear," *Journal of Applied Mechanics*, Vol. 81, pp. 285–291 (1959).

(3) Joseph Marin, "Correlations Between Transverse and Torsional Shear Creep Deformations," *WADC Technical Report 57-755*, Wright Air Development Center, June, 1959.

(4) P. J. Vawter, G. J. Guarnieri, L. A. Yerkovich, and G. Derrick, "Investigation of the Compressive, Bearing and Shear Creep-Rupture Properties of Aircraft Structural

Metals and Joints at Elevated Temperatures," *WADC Technical Report 54-270*, Parts 1, 2, and 3, 1956.

(5) E. G. Bodine, R. L. Carlson, and G. K. Manning, "Interaction of Bearing and Tensile Loads on Creep Properties of Joints," *NACA Technical Note 3758*, National Advisory Committee for Aeronautics, Oct., 1956.

(6) L. Mordfin and A. C. Legate, "Creep Behavior of Structural Joints of Aircraft Materials Under Constant Loads and Temperatures," *NACA Technical Note 3842*, Jan. 1957.

DISCUSSION

MR. R. B. CLAPPER.[1]—The paper on "Determination of Compressive, Bearing, and Shear Creep of Sheet Metals" by Messrs. Kattus and Lessley[2] showed some interesting bearing and creep deformation data. Mr. Marin's interesting analytical approach to the problem of creep deformation of joints has considered many parameters, including compressive and bearing creep deformation. Does he now plan to refine his analysis by comparison with these and other pertinent data which may have been obtained experimentally since the work of this paper was completed?

MR. J. MARIN (*author*).—The comparison of experimental results with the theoretical predictions would be an important further study that should be made. The author hopes to do this in the near future.

MR. EDWARD L. HORNE.[3]—I would also like to compliment Mr. Marin on his paper. Our (ASD) thinking in proposing this work a few years ago, at which time the mechanical equation of state was in vogue in the aircraft industry, was that this study would provoke a considerable amount of controversy and difference of opinion in this area, so that some investigators would be motivated to tackle this problem experimentally with the idea of conducting the extensive creep testing necessary to do it under the approach suggested by Mr. Clapper.

Some of the bearing creep and shear creep data obtained on ASD programs were used for multiple riveted joints by Leonard Mordfin of the National Bureau of Standards and he obtained very good correlation by using those two types of data. There has also been effort on the relationship between tensile and compressive creep so that some of the assumptions in trying to take the tensile creep and predict a solution to the entire problem from it, and the necessary simplifying assumptions which must be made to do so, indicate that this is a reasonable approach to prediction of joint creep behavior; as Mr. Marin has indicated, it is a complex problem.

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[2] See p. 920.

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CREEP AND RUPTURE PROPERTIES OF FIVE COPPER-BASE CASTING ALLOYS*

By Donald P. Moon[1] and Ward F. Simmons[1]

Synopsis

A creep evaluation was conducted on cast specimens of 76 Cu, 2½ Sn, 6½ Pb, 15 Zn, 65,000-psi manganese bronze, 110,000 psi manganese bronze, 20 per cent nickel silver, and 81-4-15 silicon brass alloys at selected temperatures between 250 and 550 F. The duration of the longest tests at each temperature was about 2000 to 3000 hr.

The creep and rupture data were correlated with elevated-temperature, short-time tensile data by means of the ASME Boiler and Pressure Vessel Code criteria. Creep data are also presented in the form of design curves and isochronous stress-strain curves. The stability of these alloys during creep exposure was determined by means of tension tests and metallographic examination after creep exposure.

The 76-2½-6½-15 alloy has high creep strength, relative to its short-time tensile strength, up to about 520 F, and its stability is good. The 65,000-psi manganese bronze is limited by its loss of creep strength above 290 F. The prolonged application of the 110,000-psi manganese bronze is limited above 350 F because of the sharp decline of its creep strength and its poor stability above that temperature. For the same reason, the long-time use of the 81-4-15 silicon brass should be restricted to 450 F or lower. Creep in the 20 per cent nickel silver is insignificant up to 550 F; the melting of a lead phase in this alloy prohibits its application at higher temperatures.

This paper presents creep and creep-rupture properties for the five cast copper-base alloys discussed in a paper (**1**)[2] presented at the 1960 ASTM Annual Meeting. The two papers together constitute the second portion of a continuing program to provide accurate measurements for mechanical and physical properties of sound test bars from good-quality melts of commercial alloys.

Data were reported earlier (**2**,**3**) for a group of three casting alloys, identified as 2A, 3A, and 4A by ASTM Specification B 30 – 59[3] for ingots and by their trade designations, Navy "M," 80-10-10, and 85-5-5-5, respectively.

The five alloys for which creep and creep-rupture data are reported in the present paper are identified as 5B, 8A, 8C, 11A, and 13B by ASTM Specification B 30 – 59.[3] The casting specifications are ASTM B 145 – 52, Alloy

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* Presented at the Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] Principal Metallurgist and Division Chief, respectively, Metallurgical Department, Battelle Memorial Inst., Columbus, Ohio.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

[3] Standard Specification for Copper-Base Alloys in Ingot Form for Sand Castings (B 30 – 59), 1959 Supplement to Book of ASTM Standards, Part 2, p. 1.

5B[4]; B 147 – 52, Alloys 8A and 8C[5]; B 149 – 52, Alloy 11A[6]; and B 198 – 58, Alloy 13B.[7] These alloys are referred to in this paper as 76Cu-2½Sn-6½Pb-15Zn, 65,000-psi manganese bronze, 110,000-psi manganese bronze, 20 per cent nickel silver, and 81-4-15 silicon brass, respectively.

TABLE I.—AVERAGE CHEMICAL ANALYSES OF CASTINGS TESTED.

Element	Chemical Analysis, per cent				
	76-2½-6½-15 Alloy	Manganese Bronze		20 per cent Nickel Silver	81-4-15 Silicon Brass
		65,000 psi	110,000 psi		
Copper	75.8	57.9	61.9	64.0	80.7
Tin	2.7	0.26	....	4.0	<0.01
Lead	5.6	0.18	....	4.1	<0.01
Zinc	14.8	39.0	25.4	6.6	15.0
Iron	0.26	1.02	3.1	0.75	0.11
Antimony	0.13	....	....	0.14	....
Nickel	0.66	0.08	....	20.4	0.01
Aluminum	....	1.07	5.8	....	....
Manganese	....	0.50	3.8	0.03	0.01
Silicon	....	....	....	....	4.12
Sulfur	0.05	....	....	....	....

Table I gives the range of chemical composition for the castings tested. The chemical analyses for all castings were well within the ASTM specification requirements.

It should be pointed out that alloys with many variations in composition may meet a given ASTM specification for chemical composition. The variations in composition could result in marked changes in mechanical properties. Therefore, the specific compositions of the five types of alloys used in the investigation were intended to be representative of the ones that are used widely by the industry.

Details of the foundry practice used in preparing and establishing the quality of the melts were given in an earlier paper (1). Specimens for the creep-testing program were machined from either a double-horizontal full-web test-bar casting or from a keel-block casting. The keel-block casting was used for the two manganese bronzes, which are considered to be high-shrinkage alloys.

The program on creep and creep-rupture testing was carried out within the temperature range of 250 to 550 F. The test temperatures were selected on the basis of the creep strengths exhibited by the various alloys as the evaluation program developed. An attempt was made to select the highest temperature at which useful data for service application might be obtained for each alloy. The specific test temperatures were as follows:

Alloy	Temperature, deg Fahr
76-2½-6½-15	350, 450, 500,[a] 550
65,000 psi manganese bronze	250,[a] 350, 450
110,000 psi manganese bronze	250,[a] 300,[a] 350, 450
20 per cent nickel silver	450,[a] 550[a]
81-4-15 silicon brass	350,[a] 400,[a] 450, 550

[a] Creep tests only.

The stress to produce a creep rate of 0.00001 per cent per hr and the stress to produce rupture in 100,000 hr are used by the ASME Boiler and Pressure Vessel Code Committee as a basis for establishing maximum allowable stresses for non-ferrous materials in the temperature range where significant creep occurs. The present investigation was directed toward the determination of the creep and rupture strengths at the temperature shown above and the correlation of these data with the elevated-temperature, short-time tensile data.

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[4] Standard Specification for Leaded Red Brass and Leaded Semi-Red Brass Sand Castings (B 145 – 52), 1958 Book of ASTM Standards, Part 2, p. 245.

[5] Standard Specification for High-Strength Yellow Brass (Manganese Bronze) and Leaded High-Strength Yellow Brass (Leaded Manganese Bronze) Sand Castings (B 147 – 52), 1958 Book of ASTM Standards, Part 2, p. 253.

[6] Standard Specification for Leaded Nickel Brass (Leaded Nickel Silver) and Leaded Nickel Bronze (Leaded Nickel Silver) Sand Castings (B 149 – 52), 1958 Book of ASTM Standards, Part 2, p. 263.

[7] Standard Specification for Silicon Bronze and Silicon Brass Sand Castings (B 198 – 58), 1958 Book of ASTM Standards, Part 2, p. 344.

Some designers are now requesting data on the stresses to produce some small amount of strain in a given time, for example, 0.1 or 0.2 per cent plastic strain in 1000 hr. These data are presented in the form of design curves, showing the time to reach designated amounts of plastic strain or rupture at various stresses, and by isochronous stress-strain curves. Pertinent details on the preparation and use of the design curves, isochronous stress-strain curves, and ASME maximum-allowable stress curves were given in a previous paper (3).

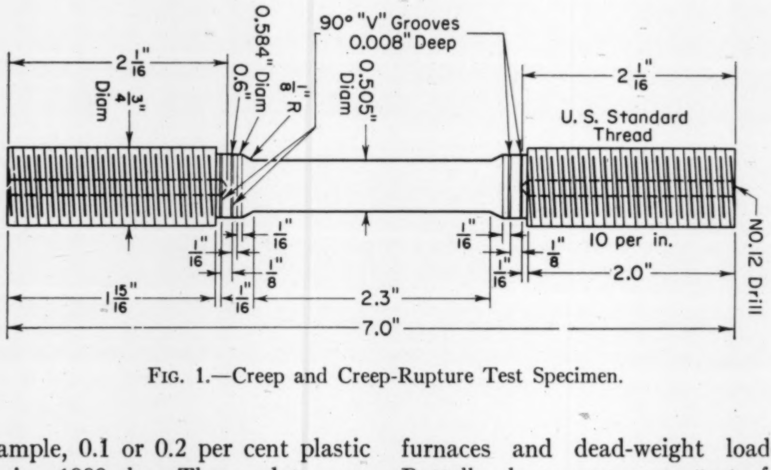


FIG. 1.—Creep and Creep-Rupture Test Specimen.

The stability of these alloys during creep was determined by measuring their retained room-temperature tensile properties after creep exposure and by metallographic examination of creep-tested specimens. The results of this study are included in this paper.

EXPERIMENTAL WORK

The creep and creep-rupture tests were conducted in accordance with ASTM Recommended Practice E 139 - 58 T, except that closer temperature control than specified in E 139 - 58 T was maintained. The specimen used in these tests is shown in Fig. 1. Its length is 7 in., diameter at the reduced section 0.505 in., and gage length 2.3 in. Specimens were heated in chromel-wound furnaces and dead-weight loaded in Battelle lever-arm creep-test frames. Strain was measured optically by means of platinum-strip extensometers and a 20-power filar micrometer microscope. The smallest division on the filar eyepiece is 0.00005 in. which, with a specimen of 2.3-in. gage length, is equal to 0.0022 per cent.

The test equipment and procedure are described in detail in a previous paper (3).

TEST RESULTS

The detailed creep and creep-rupture data for the five alloys are given in

Tables II to VI. The creep rates and rupture times listed in these tables are plotted against stress in Fig. 2 for all the alloys except the 20 per cent nickel silver. The stresses for creep in the nickel-silver alloy were very high, and only two tests were conducted at each temperature. Consequently, the data for this alloy were too limited for this type of plot.

TABLE II.—DETAILED CREEP AND CREEP-RUPTURE DATA FOR THE 76 Cu-2½ Sn-6½ Pb-15 Zn ALLOY.

Temperature, deg Fahr	Stress, psi	Duration, hr	Initial Strain, per cent	Total Strain, per cent	Contraction Upon Release of Load, per cent	Creep Rate, per cent per hr		Transition to Third-Stage Creep		Elongation, per cent	Reduction of Area, per cent
						Second Stage	Final	Time, hr	Plastic Strain, per cent		
350....	30 000	29.9^{a}	...	...	...	...	...	...	...	24.4	19.3
350....	25 000	527^{a}	16.95	...	...	0.0037	...	...	...	18.7	16.6
350....	23 000	1874^{a}	11.04	...	...	0.00094	...	...	...	16.6	20.4
350....	15 000	3291	2.371	2.807	0.139	0.000055	0.000055	>3291	...	...	...
350....	10 000	1968	0.181	0.217	0.080	0.000003	0.000003	>1968	...	...	...
450....	25 000	0.8^{a}	18.3	...	...	...	...	...	...	20.7	17.6
450....	20 000	5.8^{a}	7.55	...	...	0.16	...	...	...	11.0	17.1
450....	18 000	177^{a}	4.59	...	...	0.008	...	...	...	9.3	10.2
450....	15 000	1148^{a}	1.59	...	...	0.0008	...	...	...	6.3	13.5
450....	13 000	2309	0.625	1.277	0.120	0.00011	0.00011	>2309	...	...	...
450....	12 000	2347	0.444	0.703	0.107	0.000035	0.000035	>2347	...	...	...
450....	10 000	1849	0.194	0.285	0.087	0.00002	0.00002	>1849	...	...	...
500....	7 000	2086	0.067	0.143	0.059	0.000017	0.000017	>2086	...	...	...
550....	13 000	36.7^{a}	0.679	...	...	0.04	...	24	1.9	4.1	9.3
550....	12 000	112^{a}	0.319	...	...	0.0082	...	80	1.1	3.7	4.7
550....	10 000	924^{a}	0.177	...	...	0.0003	...	450	0.32	2.8	2.9
550....	9 600	2225^{a}	0.170	...	...	0.00021	...	1000	0.41	1.6	3.6
550....	7 000	2810	0.071	0.294	0.060	0.000055	0.000055	>2810	...	...	...
550....	4 000	2832	0.036	0.112	0.034	0.000018	0.000018	>2832	...	...	...

^{a} Rupture time.

The creep strengths for creep rates of 0.00001 and 0.0001 per cent per hr and the strengths for rupture in 100, 1000, 10,000, and 100,000 hr are summarized in Table VII. The 10,000 and 100,000-hr rupture strengths were extrapolated from tests of about 2000 hr duration for four of the alloys. The 65,000-psi manganese bronze alloy was evaluated in an earlier part of the program, in which relatively short-time rupture data were sought, and somewhat longer extrapolations were required. The extrapolation was done by applying the "mirror-image" relationship previously used by the authors (4). This relationship, attributed to J. G. Kaufman (5), takes note of the fact that, at stresses where both creep and rupture data are available, the stress-versus-rupture time curve is often a near-mirror image of the stress-versus-creep rate curve. The extrapolation of relatively short-time rupture data in this manner is considered more reliable and conservative than straight-line extrapolation of the stress-versus-rupture time curves or extrapolation by means of parameters, such as the well-known Larson-Miller parameter (6).

TABLE III.—DETAILED CREEP AND CREEP-RUPTURE DATA FOR THE 65,000-PSI MANGANESE BRONZE ALLOY.

Temperature, deg Fahr	Stress, psi	Duration, hr	Initial Strain, per cent	Total Strain, per cent	Contraction Upon Release of Load, per cent	Creep Rate, per cent per hr		Transition to Third-Stage Creep		Elongation, per cent	Reduction of Area, per cent
						Second Stage	Final Stage	Time, hr	Plastic Strain, per cent		
250......	55 000	6.1[a]	...	...	...	...	...	...	...	63.3	58.5
250......	48 000	211	...	...	...	...	...	...	...	...	...
250......	35 000	2 021	0.920	1.428	0.317	0.000090	0.000090	>2021	...	...	...
250......	30 000	2 089	0.437	0.536	0.261	0.000020	0.000020	>2089	...	...	...
250......	20 000	2 013	0.180	0.193	0.166	nil	nil	>2013	...	...	...
350......	45 000	4.1[a]	5.5	...	...	6.8	...	...	...	54.8	67.0
350......	40 000	48[a]	2.3	...	...	0.4	...	26	15.7	64.9	70.6
350......	35 000	225[a]	0.67	...	...	0.12	...	35	6.0	85.0	71.4
350......	30 000	682[a]	0.38	...	...	0.035	...	>250	>10.0	97.8	75.9
350......	20 000	1 585	0.176	7.8	0.200	0.0040	0.0059	900	3.6	...	...
350......	15 000	1 995	0.135	2.5	0.146	0.0011	0.00135	1600	1.8	...	...
350......	12 000	2 206	0.095	0.813	0.103	0.00029	0.00038	1200	0.3	...	...
350......	8 000	10 003	0.069	0.750	0.071	0.000035	0.000052	4200	0.13	...	...
450......	35 000	12.6[a]	4.0	...	...	...	...	...	...	86.5	73.0
450......	30 000	8.9[a]	...	...	...	...	...	...	...	73.4	66.7
450......	12 000	543	0.122	91.0	...	0.04	0.16	160	10.0	...	...
450......	8 000	1 873	0.082	12.3	0.11	0.0054	0.0054	>1873	...	...	...
450......	5 000	2 785	0.046	0.982	0.044	0.00012	0.00012	>2785	...	...	...
450......	3 000	2 023	0.031	0.190	0.024	0.000055	0.000055	>2023	...	...	...
450......	2 000	3 046	0.013	0.103	0.025	0.000015	0.000015	>3046	...	...	...

[a] Rupture time.

TABLE IV.—DETAILED CREEP AND CREEP-RUPTURE DATA FOR THE 110,000-PSI MANGANESE BRONZE ALLOY.

Temperature, deg Fahr	Stress, psi	Duration, hr	Initial Strain, per cent	Total Strain, per cent	Contraction Upon Release of Load, per cent	Creep Rate, per cent per hr		Transition to Third-Stage Creep		Elongation, per cent	Reduction of Area, per cent
						Second Stage	Final	Time, hr	Plastic Strain, per cent		
250.....	60 000	1417	0.470	0.545	0.450	0.000015	0.000015	>1417	...	...	...
250.....	50 000	1177	0.429	0.442	0.433	0.000004	0.000004	>1177	...	...	...
300.....	35 000	1678	0.283	0.318	0.280	0.000015	0.000015	>1678	...	...	...
350.....	70 000	27.4[a]	0.702	...	...	0.38	...	6	3.2	26.2	45.2
350.....	60 000	129[a]	0.563	...	...	0.081	...	...	...	32.8	71.5
350.....	48 000	583[a]	0.429	...	...	0.007	...	90	0.70	29.2	51.7
350.....	40 000	2246[a]	0.332	...	...	0.0013	...	270	0.39	31.3	56.8
350.....	30 000	2690	0.238	0.746	0.252	0.00012	0.00028	1300	0.29	...	...
350.....	22 000	2350	0.210	0.279	0.182	0.000020	0.000020	>2350	...	...	...
450.....	50 000	2.0[a]	0.772	...	...	5.5	...	0.5	3.1	53.9	57.8
450.....	30 000	29.3[a]	0.250	...	...	0.13	...	...	...	70.0	67.7
450.....	17 000	240[a]	0.135	...	...	0.015	...	25	0.46	68.0	74.2
450.....	12 000	3674	0.106	18.38	...	0.0042[b]	0.0042	>3674	...	...	...
450.....	5 000	3214	0.038	0.548	0.043	0.00010[b]	0.00010	>3214	...	...	...
450.....	3 000	3380	0.020	0.275	0.028	0.000057[b]	0.000057	>3380	...	...	...
450.....	1 000	3167	0.013	0.122	0.010	0.000020[b]	0.000020	>3167	...	...	...

[a] Rupture time.

[b] These specimens exhibited a very low creep rate for a short time at the beginning of the test and true second stage creep later in the test. Only the latter value is reported herein.

TABLE V.—DETAILED CREEP AND CREEP-RUPTURE DATA FOR THE 20 PER CENT NICKEL SILVER ALLOY.

Temperature, deg Fahr	Stress, psi	Duration, hr	Initial Strain, per cent	Total Strain, per cent	Contraction Upon Release of Load, per cent	Creep Rate, per cent per hr		Transition to Third-Stage Creep		Elongation, per cent	Reduction of Area, per cent
						Second Stage	Final	Time, hr	Plastic Strain, per cent		
450....	34 000	2500	12.06	12.20	0.304	0.000015	0.000015	>2500	...	...	...
450....	30 000	2814	7.24	7.32	0.249	0.000005	0.000005	>2814	...	...	...
550....	23 000	312[a]	0.656	0.671[b]	...	...	0.00002	...	...	0.8	1.5
550....	20 000	1518	0.281	0.305	0.151	0.000001	0.000001	>1518	...	...	...
650....	15 000	0.6[c]	0.568	...	...	...	...	...	...	64.6	68.3
650....	10 000	3.5[c]	0.122	...	...	...	...	...	...	77.0	67.4

[a] Specimen failed prematurely when jarred by rupture of specimen in adjoining unit.

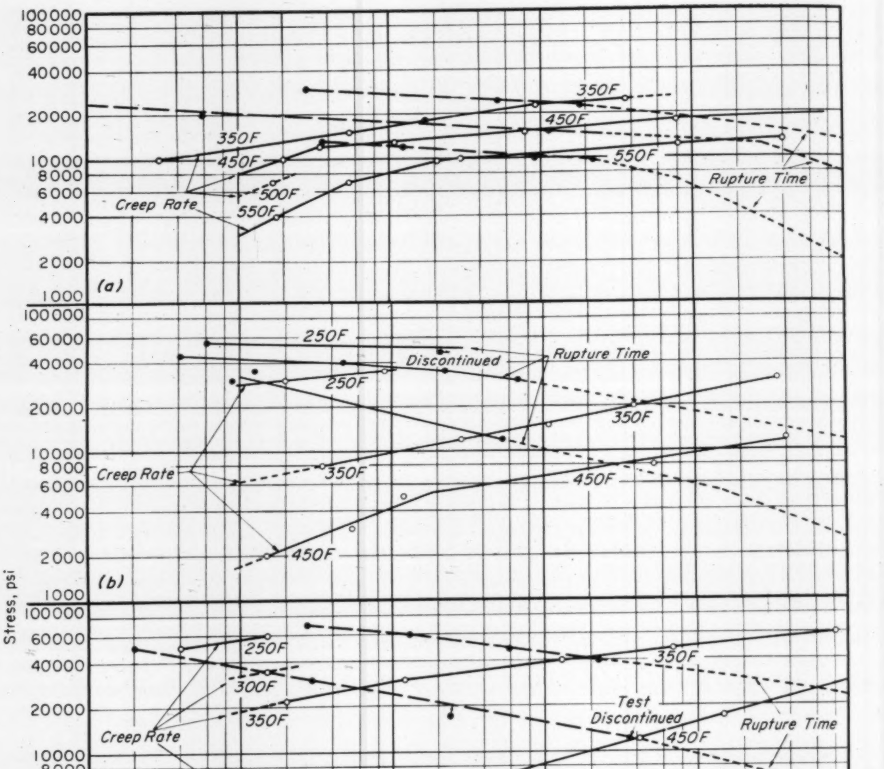
[b] Strain immediately prior to failure.

[c] Rupture time.

TABLE VI.—DETAILED CREEP AND CREEP-RUPTURE DATA FOR THE 81-4-15 SILICON BRASS ALLOY.

Temperature, deg Fahr	Stress, psi	Duration, hr	Initial Strain, per cent	Total Strain, per cent	Contraction Upon Release of Load, per cent	Creep Rate, per cent per hr		Transition to Third-Stage Creep		Elongation, per cent	Reduction of Area, per cent
						Second Stage	Final	Time, hr	Plastic Strain, per cent		
350......	40 000	1901	1.322	2.080	0.375	0.00016	0.00016	>1901	...	...	...
350......	30 000	2616	0.542	0.707	0.247	0.000016	0.000016	>2616	...	...	...
400......	20 000	2973	0.216	0.323	0.165	0.000017	0.000017	>2973	...	...	...
450......	50 000	14.4[a]	4.33	...	...	0.40	...	>9	...	13.1	16.5
450......	40 000	390[a]	1.09	...	...	0.024	...	140	5.2	13.1	27.9
450......	35 000	1793[a]	0.739	...	...	0.0047	...	230	1.8	13.8	17.9
450......	30 000	1057	0.572	2.85	0.28	0.0016	0.0025	550	1.5	...	...
450......	20 000	3263	0.225	0.735	0.181	0.000115	0.000115	>3263	...	...	...
450......	13 000	3401	0.111	0.260	0.112	0.000017	0.000017	>3401	...	...	...
550......	40 000	3.2[a]	1.72	...	...	3.1	...	...	...	12.6	14.8
550......	30 000	40.3[a]	0.548	...	...	0.15	...	...	...	12.3	19.4
550......	15 000	1588[a]	0.141	...	...	0.0016	...	600	1.2	6.6	11.4
550......	8 000	2351	0.072	0.554	...	0.00014	0.00017	1700	0.40	...	...
550......	4 000	3000	0.034	0.205	0.033	0.000035	0.000043	2300	0.14	...	...
550......	2 000	3194	0.025	0.107	0.026	0.000015	0.000015	>3194	...	...	...

[a] Rupture time.



(*a*) 76Cu-2½Sn-6½Pb-15Zn alloy. (*b*) 65,000 psi manganese bronze alloy. (*c*) 110,000 psi manganese bronze alloy. (*d*) 81-4-15 silicon brass alloy.

FIG. 2.—Stress Versus Creep Rate and Rupture Time.

TABLE VII.—SUMMARY OF CREEP AND RUPTURE STRENGTHS.

Alloy	Temperature, deg Fahr	Creep Strength, psi		Rupture Strength, psi			
		Creep Rate of 0.00001 per cent per hr	Creep Rate of 0.0001 per cent per hr	100 hr	1000 hr	10,000 hr	100,000 hr
76-2½-6½-15	350	11 900	16 400	27 500	24 000	18 000[a]	12 800[a]
	450	8 000	13 000	17 700	15 200	12 900[a]	7 800[a]
	500	5 600	...	...	...	...	...
	550	3 000	8 000	12 100	10 100	6 300[a]	2 000[a]
65,000-psi manganese bronze	250	28 000	35 200	>50 000	...	...	...
	350	6 200	9 600	37 000	28 000[a]	18 000[a]	11 600[a]
	450	1 700	4 200	18 100	10 200[a]	5 900[a]	2 500
110,000-psi manganese bronze	250	56 500	...	...	...	...	...
	300	32 500	...	...	...	...	...
	350	19 000	29 000	61 000	45 000	33 000[a]	22 500[a]
	450	0 500	4 400	24 000	15 500	9 400[a]	5 000[a]
20 per cent nickel silver	450	32 500	...	...	...	...	...
	550	22 200	...	...	...	...	...
81-4-15 silicon brass	350	28 000	38 000	...	...	...	...
	400	18 000	...	...	...	...	...
	450	11 000	19 300	44 000	36 500	27 200[a]	19 000[a]
	550	1 400	6 800	25 200	16 300	9 300[a]	3 400[a]

[a] Extrapolated.

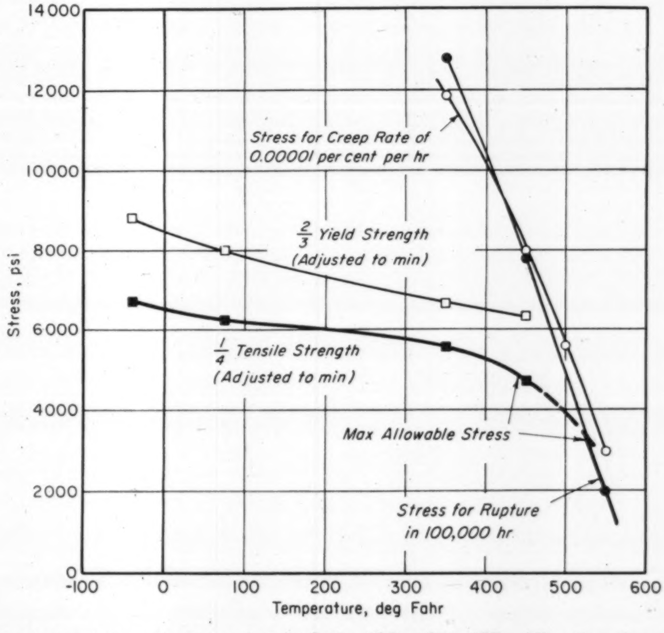
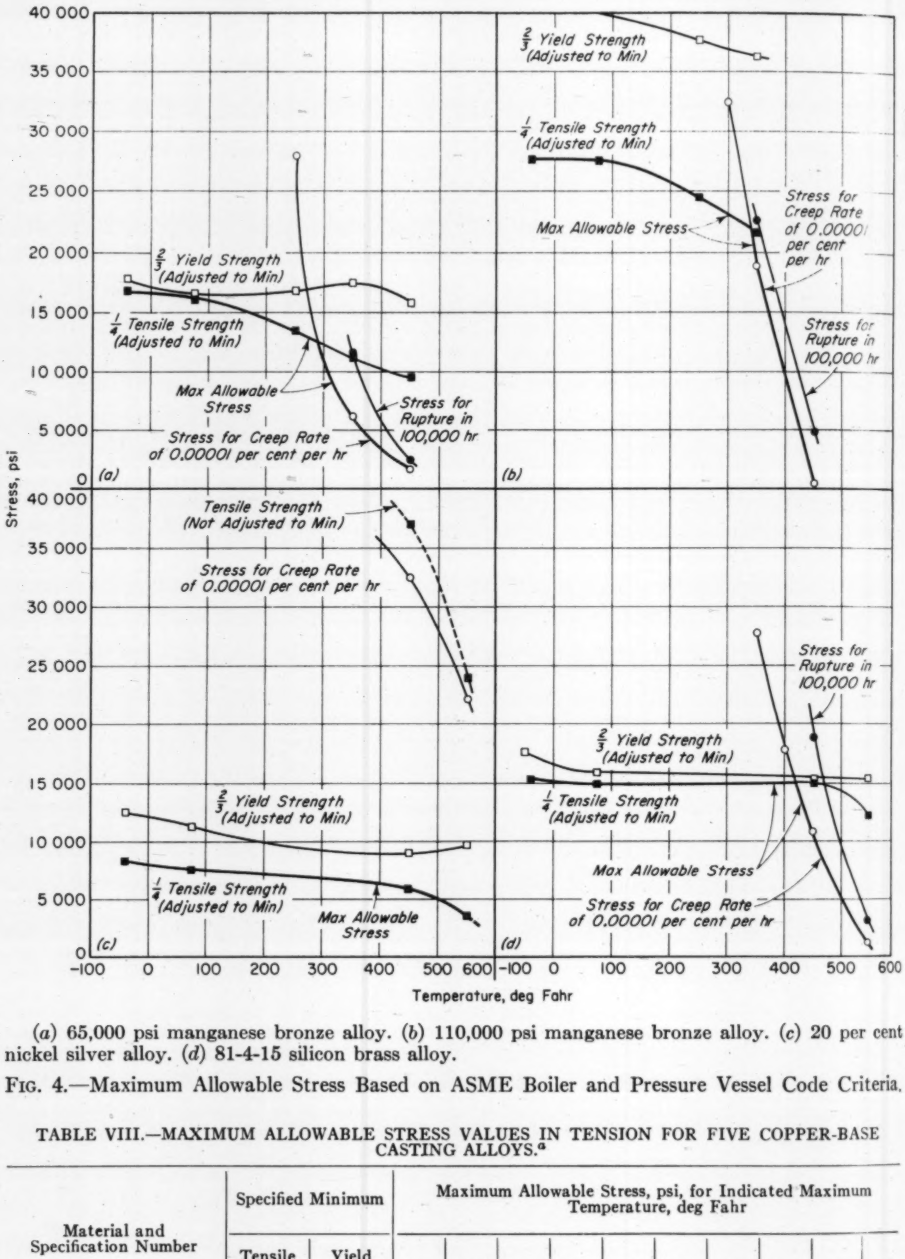


FIG. 3.—Maximum Allowable Stress for the 76Cu-2½Sn-6½Pb-15Zn Alloy Based on ASME Boiler and Pressure Vessel Code Criteria.



(*a*) 65,000 psi manganese bronze alloy. (*b*) 110,000 psi manganese bronze alloy. (*c*) 20 per cent nickel silver alloy. (*d*) 81-4-15 silicon brass alloy.

FIG. 4.—Maximum Allowable Stress Based on ASME Boiler and Pressure Vessel Code Criteria.

TABLE VIII.—MAXIMUM ALLOWABLE STRESS VALUES IN TENSION FOR FIVE COPPER-BASE CASTING ALLOYS.[a]

Material and Specification Number	Specified Minimum		Maximum Allowable Stress, psi, for Indicated Maximum Temperature, deg Fahr									
	Tensile Strength, psi	Yield Strength, psi	100	150	200	250	300	350	400	450	500	550
76-2½-6½-15, B 145 - 52	25 000	12 000	6 100	6 000	5 900	5 800	5 700	5 500	5 200	4 700	3 900	2 000
65,000 psi manganese bronze, B 147 - 52	65 000	25 000	16 000	15 300	14 500	13 500	11 000	6 200	3 400	1 700	...	...
110,000 psi manganese bronze, B 147 - 52	110 000	60 000	27 300	26 700	25 700	24 400	23 000	19 000	...	...	...	...
20 per cent nickel silver, B 149 - 52	30 000	17 000	7 300	7 200	7 000	6 900	6 700	6 600	6 300	5 800	5 000	3 600
81-4-15 silicon brass, B 198 - 58	60 000	24 000	15 000	15 000	15 000	15 000	15 000	15 000	15 000	11 000	...	...

[a] Stresses based on criteria outlined in Appendix Q of Section VIII of "Rules for Construction of Unfired Pressure Vessels," 1959.

Maximum Allowable Stresses:

The ASME Boiler and Pressure Vessel Committee has established criteria for selecting the maximum allowable stress values for non-ferrous materials. These criteria, as outlined in Appendix Q of Section VIII of "Rules for Construction of Unfired Pressure Vessels," 1959, are as follows:

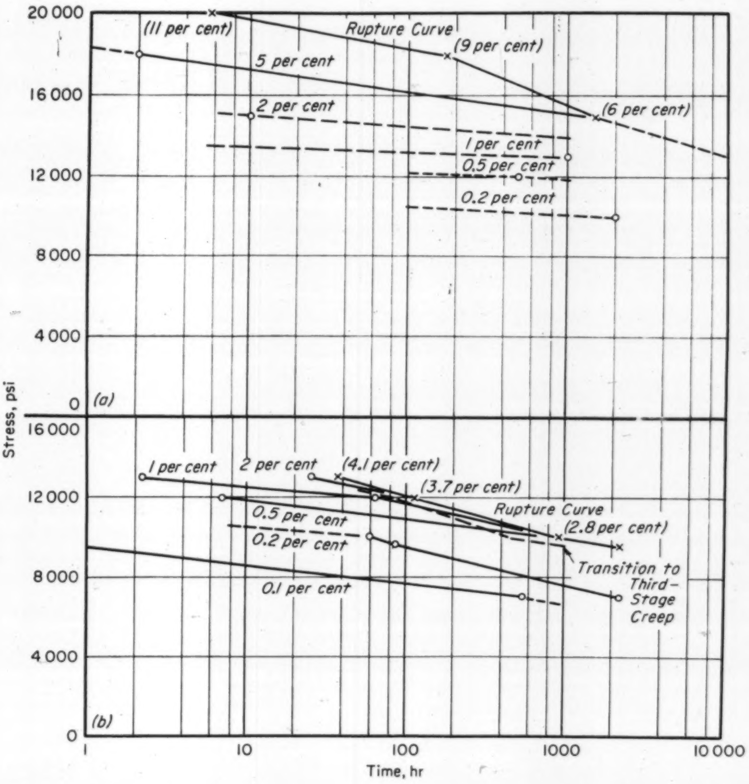
"Except for bolting materials, the maximum allowable stress values may be determined as the lowest of the following when the tensile and yield strengths are obtained from standard short-time tests:

(*a*) One-fourth of the tensile strength as adjusted to minimum.[8]

(*b*) Two-thirds of the yield strength (as defined in the material specification) as adjusted to minimum.[8]

(*c*) The stress producing a secondary creep rate of 0.1 CRU (creep rupture units) or 0.1 per cent in 10,000 hr [0.00001 per cent per hr].

(*d*) The stress producing rupture in 100,000 hr."



(*a*) At 450 F. (*b*) At 550 F.

FIG. 5.—Stress Versus Time for Various Amounts of Plastic Strain and Rupture for 76Cu-2½Sn-6½Pb-15Zn Alloy.

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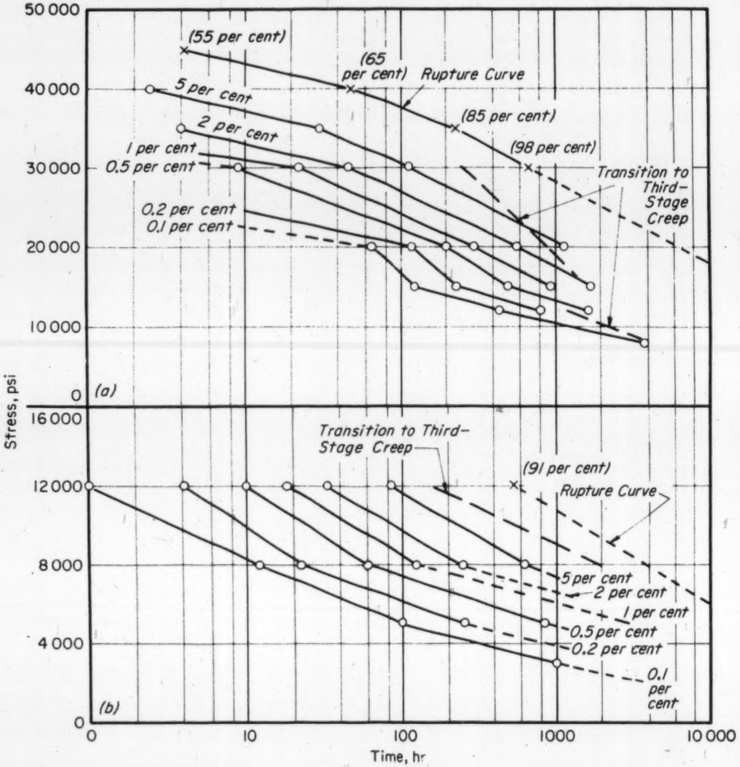
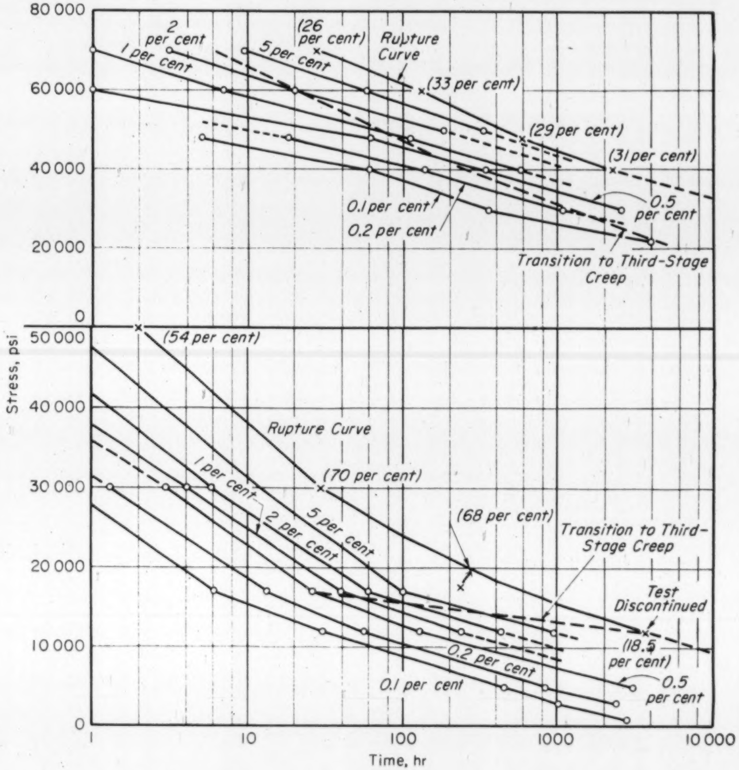
[8] Strength is adjusted to minimum by multiplying the results at each temperature by the ratio of (minimum specified tensile or yield strength) to (actual room-temperature tensile or yield strength for the lot of material under test).



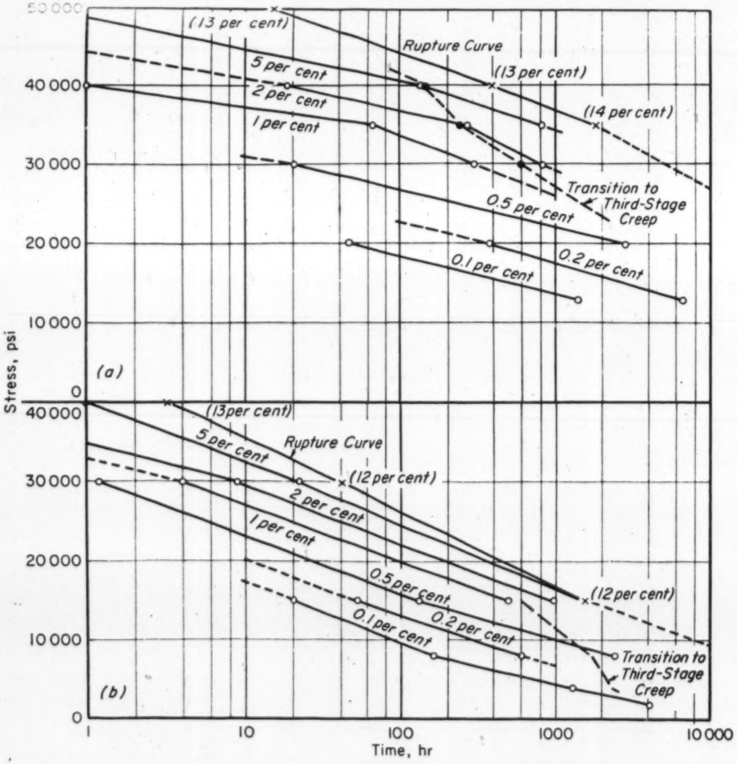
(*a*) 350 F. (*b*) 450 F.

FIG. 6.—Stress Versus Time for Various Amounts of Plastic Strain and Rupture for 65,000 psi Manganese Bronze Alloy.



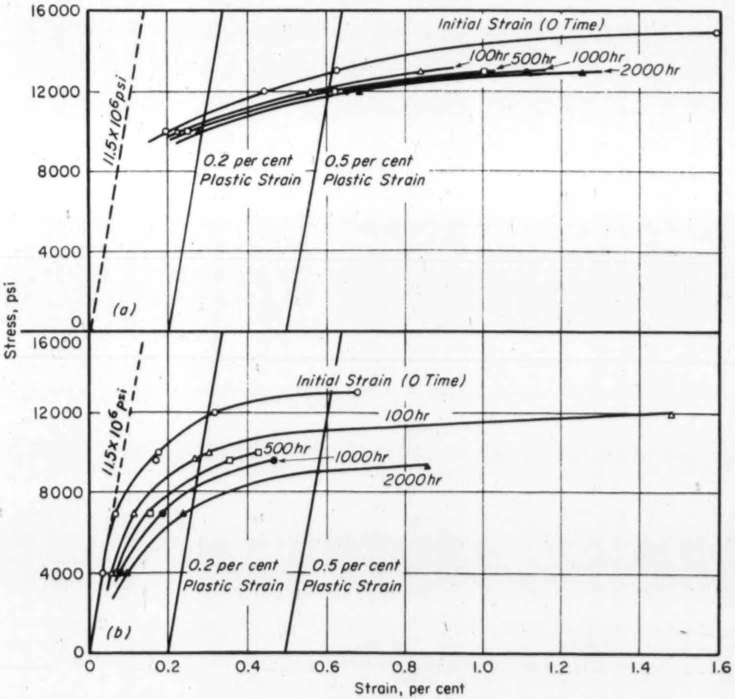
(*a*) 350 F. (*b*) 450 F.

FIG. 7.—Stress Versus Time for Various Amounts of Plastic Strain and Rupture for the 110,000 psi Manganese Bronze Alloy.



(*a*) 450 F. (*b*) 550 F.

FIG. 8.—Stress Versus Time for Various Amounts of Plastic Strain and Rupture of 81-4-15 silicon brass.

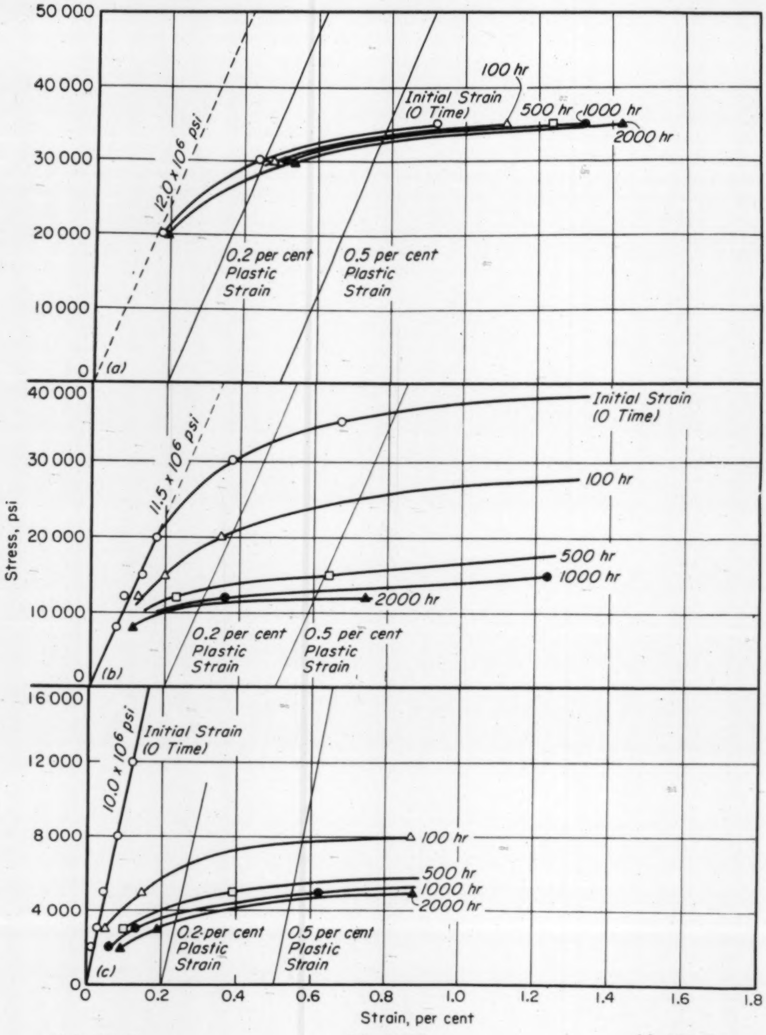


(*a*) 450 F. (*b*) 550 F.

FIG. 9.—Isochronous Stress-Strain Curves for a 76Cu-2½Sn-5½Pb-15Zn Alloy.

(Note: the 1956 code specified four-fifths of the stress for rupture in 100,000 hr.)

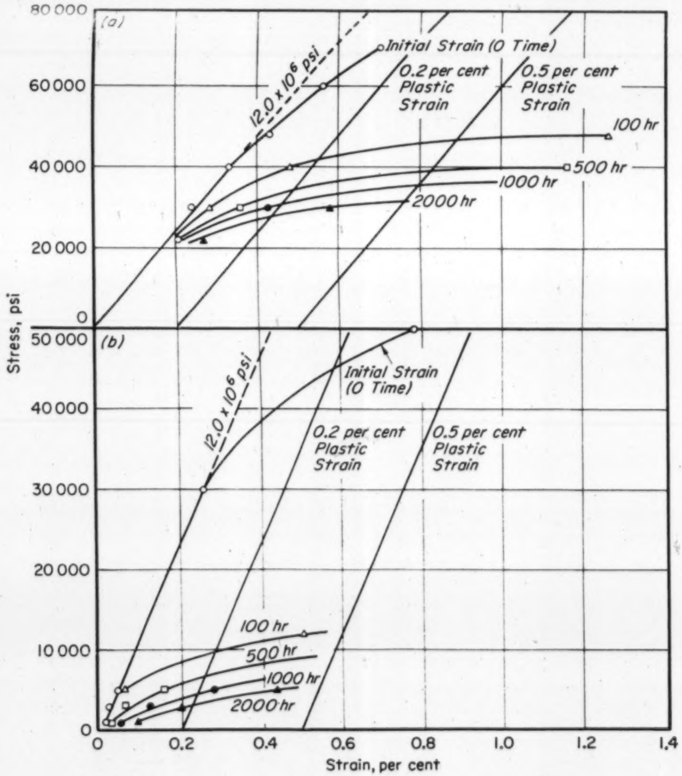
The data presented in this paper, together with the elevated-temperature, short-time tensile data in the companion paper (1), were used to determine the maximum allowable stresses in the manner prescribed by the ASME code requirements. The results are shown in Figs. 3 and 4 as stress-versus-temperature curves.



(a) 250 F. (b) 350 F. (c) 450 F.

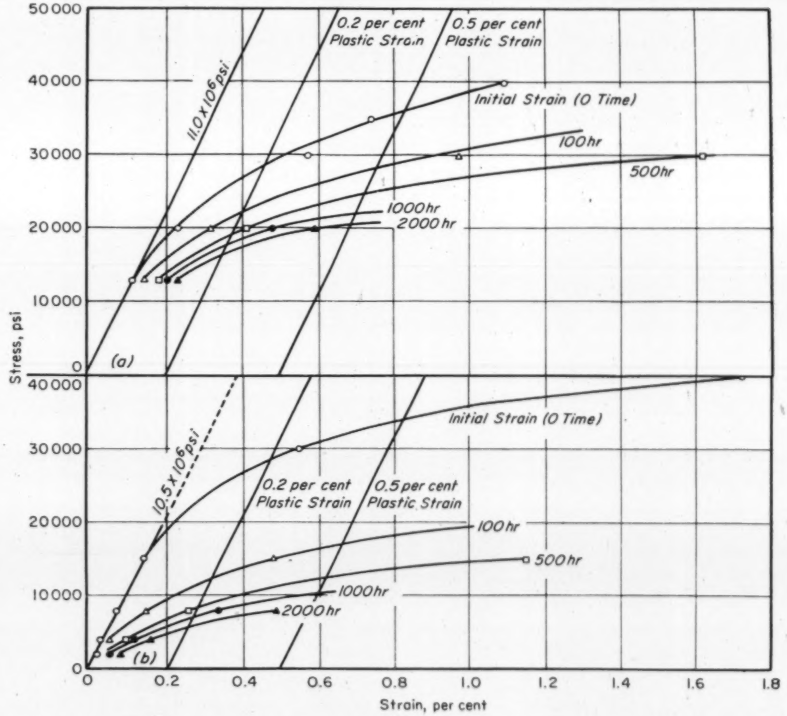
FIG. 10.—Isochronous Stress-Strain Curves for the 65,000 psi Manganese Bronze.

These curves show the over-all relationship between short-time and long-time strength properties, and they are helpful in determining the temperature at which creep becomes significant. They do not, however, indicate the degree to which an alloy may be damaged



(*a*) 350 F. (*b*) 450 F.

FIG. 11.—Isochronous Stress-Strain Curves for the 110,000 psi Manganese Bronze.



(*a*) 450 F. (*b*) 550 F.

FIG. 12.—Isochronous Stress-Strain Curves for 81-4-15 Silicon Brass.

TABLE IX.—ROOM-TEMPERATURE TENSILE PROPERTIES OF UNBROKEN CREEP-TESTED SPECIMENS.

Alloy	Creep-Test Conditions				Yield Strength, psi		Tensile Strength, psi	Elongation in 2 in., per cent	Reduction of Area, per cent	Young's Modulus, psi	Fracture
	Temperature, deg Fahr	Stress, psi	Duration, hr	Plastic Strain, per cent	0.2 per cent offset	0.5 per cent extension					
76-2½-6½-15	Before creep testing[a]				14 000	14 900	37 400	35	29	15.2 × 10⁶	Uniformly gray, fine structure
	350	10 000	1 968	0.14	14 700	15 500	35 600	29	25	11.8	Gray, mottled with coppery granules
	450	10 000	1 849	0.20	14 700	15 500	35 600	30	26	12.6	Gray, mottled with coppery granules
	550	7 000	2 810	0.23	16 400	17 400	35 400	23[b]	19	15.1	Gray, mottled with coppery granules
65,000-psi manganese bronze	Before creep testing[a]				31 400	32 800	80 200	36	29	14.8	Uniformly golden-gray, fine structure
	350	12 000	2 206	0.71	33 900	34 600	79 100	29	26	13.7	Uniformly coppery
	350	8 000	10 003	0.68	33 000	34 600	79 300	26	28	14.9	Uniformly coppery
	450	3 000	2 023	0.16	33 400	35 000	77 800	27	28	13.8	Uniformly coppery
110,000-psi manganese bronze	Before creep testing[a]				83 100	68 600	119 300	17	20	14.2	Uniformly golden-gray, fine structure
	300	35 000	1 678	0.04	94 600	74 700	118 000	4	11	15.4	Uniformly coppery, granular
	350	22 000	2 350	0.10	93 900	73 000	109 000	3	5	14.9	Uniformly coppery, granular
	450	5 000	3 214	0.50	...	...	*c*	...	...	...	Uniformly gray, coarsely granular
	450	3 000	3 380	0.25	...	...	*d*	...	...	17.5	Uniformly gray, coarsely granular
	450	1 000	3 167	0.11	...	...	*e*	...	...	...	Uniformly gray, coarsely granular
20 per cent nickel silver	Before creep testing[a]				24 600	26 000	46 900	21	21	19.1	Dense, uniformly gray, fine structure
	550	20 000	1 518	0.15	37 400	38 400	47 000	9	11	16.4	Uniformly gray
81-4-15 silicon brass	Before creep testing[a]				27 200	30 000	68 200	18	20	15.4	Uniformly light gray, fine structure
	450	13 000	3 410	0.15	41 800	42 400	64 000	10	16	14.7	Uniformly coppery-gray, ragged
	550	4 000	3 000	0.17	47 000	45 700	56 400	1	1	14.2	Uniformly gray, somewhat columnar

[a] Average values taken from Fig. 1 of companion paper (**1**).

[b] Specimen failed outside of gage marks.

[c] Specimen failed in threaded section at a stress of 67,600 psi.

[d] Specimen failed in threaded section at a stress of 63,100 psi.

[e] Specimen failed in threaded section at a stress of 69,000 psi.

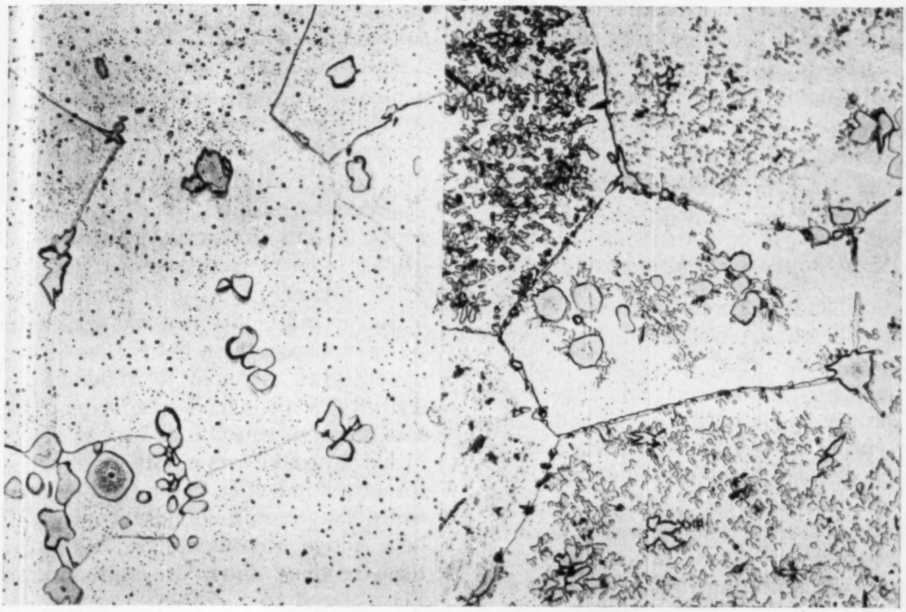


FIG. 13.—Microstructure of 11,000 psi Manganese Bronze Before and After Creep Testing for 3167 hr at 450 F and a Stress of 1000 psi (× 500). Etched in lactic-nitric acids.

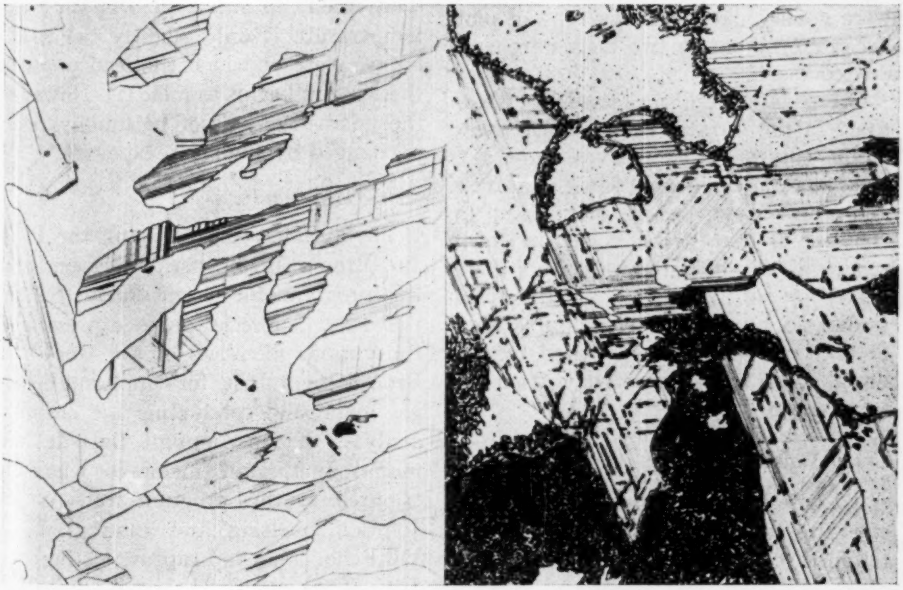


FIG. 14.—Microstructure of 81-4-15 Silicon Brass Before and After Creep Testing for 3000 hr a 550 F and a Stress of 4000 psi (× 500). Etched in $FeCl_3$-HCl.

through creep exposure. The creep stability of the alloy must also be considered in designing for prolonged elevated-temperature application. Creep stability of these alloys was taken into account in tabulating the ASME maximum allowable stress values in Table VIII.

Stress-Strain-Time Relationships:

When design data are required for small amounts of strain in specified periods of time, "design curves" and isochronous stress-strain curves have proven to be optimum methods of presenting creep data. The data in this paper are presented both ways.

The design curves in Figs. 5 to 8 show stress plotted against time for various amounts of plastic strain from 0.1 to 5 per cent and for rupture for each of the alloys except the nickel silver. In the case of the nickel-silver alloy at all test temperatures, and the other alloys at certain temperatures, sufficient data were not available for this type of plot because only one or two tests were conducted.

Creep data are also plotted in Figs. 9 to 12 in the form of isochronous stress-strain curves. These curves show the total strain upon loading to a given stress and after various periods of time, up to 2000 hr, at that stress. Plastic or creep strain can also be determined from these curves by subtracting the elastic strain (indicated by the elastic modulus line) or the load-on strain (indicated by the initial strain line), respectively, from the total strain.

Creep-Stability Studies:

To determine the effect of long-term exposure to stress and temperature on retained room-temperature tensile properties, selected creep-tested specimens were subsequently tested in short-time tension at room temperature. The results are given in Table IX.

As a result of exposure to elevated temperatures during creep, tensile yield strength increased and tensile ductility decreased in each specimen. In only two alloys was the decreased ductility considered serious. The 110,000-psi manganese bronze suffered a substantial loss of room-temperature ductility as a result of creep exposure at temperatures of 300 F and higher and was embrittled by creep exposure at 450 F. The 81-4-15 silicon brass suffered a slight loss of ductility as a result of creep exposure at 450 F and a substantial loss at 550 F. Metallographic examination of these specimens indicated the formation of an embrittling grain-boundary phase in both alloys. Representative microstructures of these alloys are reproduced in Figs. 13 and 14.

The microstructures of the 76Cu-$2\frac{1}{2}$Sn-$6\frac{1}{2}$Pb-15Zn and the 20 per cent nickel-silver alloys were also examined after creep exposure at 550 F, since this temperature is only slightly below the temperature at which the lead phase in these alloys begins to melt (1). However, the microstructures of both alloys were unchanged by the creep exposure.

Acknowledgment:

The authors wish to thank the Brass and Bronze Ingot Inst., the sponsor of this program, for permission to publish the data. Appreciation is expressed to the various members of the Brass and Bronze Ingot Inst. for supplying the ingot metal and conducting the chemical analyses of the castings. Battelle Memorial Inst. staff members who contributed to this program were W. H. Johnson, melting and casting; J. A. VanEcho, creep and rupture testing; and J. G. Kura and C. H. Lorig, advice and guidance.

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ANOMALOUS FRACTURE IN THE CREEP OF NICKEL*

By T. C. Reuther,[1] P. Shahinian,[1] and M. R. Achter[2]

Synopsis

Nickel and nickel-base alloys tested in creep at relatively high temperature and low stress sometimes fail in the fillet where stress is appreciably less than in the gage section. This behavior is observed to occur in air, but never in vacuum.

In the investigation of this anomaly, tapered nickel rods creep tested at 1200 and 1500 F fractured at the minimum diameter section in all vacuum and mildly oxidizing air tests. However, under strongly oxidizing conditions—long times in air at 1500—the fracture position was progressively displaced into heavier sections as the applied stress was reduced. Similarly, conventional specimens of different diameters creep tested at the same stress revealed that fracture may occur sooner in larger sections. Metallographic studies showed that under highly oxidizing creep conditions small sections are strengthened more rapidly than large sections by the formation of load-bearing oxide which bridges intergranular cracks. Consequently, internal cracks which remain free of oxide lead to earlier failure in the larger sections.

The anomalous fractures are explained on the basis of the relative extent of oxide-filled and oxide-free cracks in small and large sections of the specimens.

Nickel and nickel-base alloys tested in creep sometimes unaccountably fail in the fillet where the stress is appreciably lower than in the gage section. This behavior appears to be different from the type of failure at the extreme of the gage section which is generally attributed to a stress concentration. In the present case the anomalous fracture does not occur in vacuum but takes place only after creep in high-temperature air for a long time. Previous work by Shahinian and Achter has shown that environment can have a dual role (**1**,**2**).[3] Surface adsorption of oxygen can facilitate intergranular crack initiation and propagation but subsequent oxidation of the cracks can impart creep resistance which is superior to that of the original material. Widmer and Grant (**3**) have also shown that oxidation can materially increase rupture strength and the results of McHenry and Probst (**4**) as well as of Douglas (**5**) indicated that the strengthening depends on section size.

These environmental effects suggested that fracture takes place in the fillet when oxidation has prevented failure in the gage section. To investigate the mechanism of this anomalous fracture, creep-rupture tests and metallographic

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

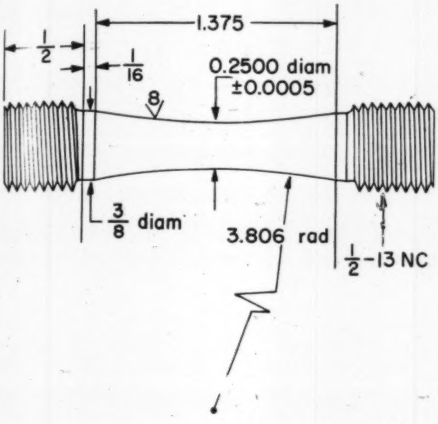


FIG. 1.—Tapered Creep-Rupture Specimen.

studies were conducted on specimens with tapered gage sections and on conventional ones of two different diameters. A mechanism based on the rupture strength of oxide layers is offered to explain the observations.

TEST PROCEDURE

In order to reproduce the fillet fracture behavior under conditions which facilitate the location of the fracture position with respect to the minimum section and which improve the accuracy of diameter measurement, a tapered gage section was employed as shown in Fig. 1. Conventional rod-type specimens 0.250 and 0.357 in. in diameter were used to separate the effects of section size and stress which are indistinguishable in tapered rods. These were machined from hot-rolled nickel of 99.8 per cent purity which contained as impurities carbon, silicon, copper, and iron. The tapered specimens were produced from one lot of material while the conventional ones were fabricated from another. Following machining, an anneal of 2 hr at 1700 F in a vacuum of 10^{-5} mm of mercury gave an average grain diameter of 0.1 mm. Creep rupture testing was performed in air with conventional constant load equipment and in a vacuum of less than 10^{-5} mm of mercury in equipment described by Shahinian (**6**).

TABLE I.—CREEP-RUPTURE RESULTS OF TAPERED NICKEL SPECIMENS WHICH SHOW THE EFFECT OF ATMOSPHERE.

Temperature, deg Fahr	Initial Stress at Minimum Diameter, psi	Atmosphere	Rupture Life, hr	Fracture Position
1200	9000	air	103	minimum diameter
		vacuum	133	minimum diameter
	5500	air	849	minimum diameter
		vacuum	1190	minimum diameter
1500	4500	air	25.5	minimum diameter
		vacuum	53.3	minimum diameter
	2500	air	220	0.158 in. from minimum diameter
		vacuum	145	minimum diameter

TEST RESULTS

Tapered Specimens:

An initial series of creep-rupture tests was run on tapered rods at 1200 and 1500 F in air and in vacuum to define the conditions under which anomalous fracture would take place (Table I). A displacement of the rupture location from the minimum section was observed only in air at a temperature and stress where the rupture life is greater than in vacuum.

A second series was run to follow more closely the relationship of rupture life to fracture location. In Fig. 2(*b*) it is evident that as test life increases, failure takes place at progressively greater distances from the minimum section. At an initial stress of 2000 psi rupture occurred after 350 hr of creep at a location where the stress was 12 per cent less than at the minimum diameter (Fig. 2(*c*)). A 24 per cent difference was observed when, after 2200 hr of creep at 1850 psi, the load was increased until fracture occurred. These differences were computed from the relationship $\left[1 - \left(\frac{d_m}{d_f}\right)^2\right]100$, where d_m and d_f are the diameters, measured after rupture, at the minimum and fracture locations, respectively. The discontinuity in slope of the stress versus rupture-life plot (Fig. 2(*a*)) indicative of pronounced oxidation strengthening has been discussed previously (1–5).

Macrographs of longitudinal sections in Fig. 3 graphically show the displacement of the fracture location and its relationship to the pattern of intergranular cracks. Of prime importance to the interpretation of these results was the observation that there were no internal cracks beyond the fracture position. It was found that the detailed study of the failure mechanism was facilitated by the presence of an incipient fracture at an equivalent position on the opposite taper, as shown clearly in Fig. 3(*c*). Microscopic examination of this area revealed that the last crack at the center line (Fig. 4) was free of oxide, those close by were partially filled, and those near the mini-

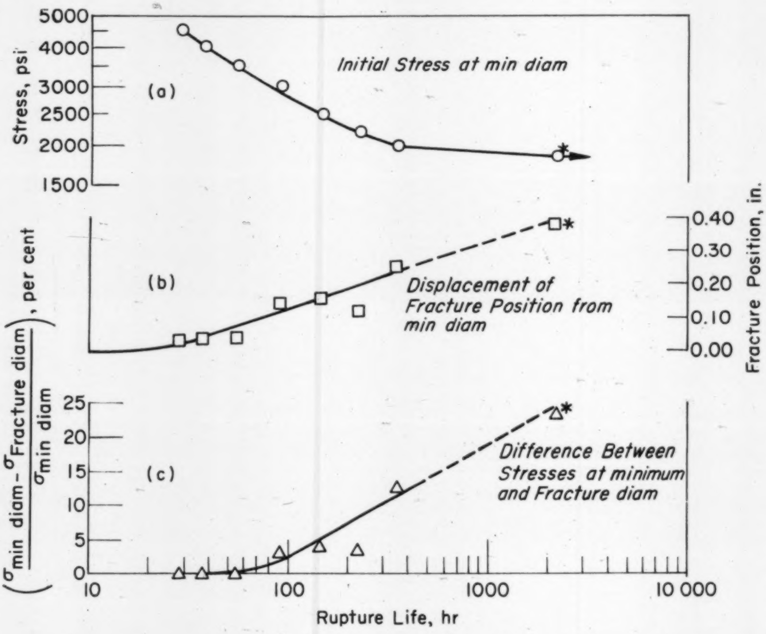
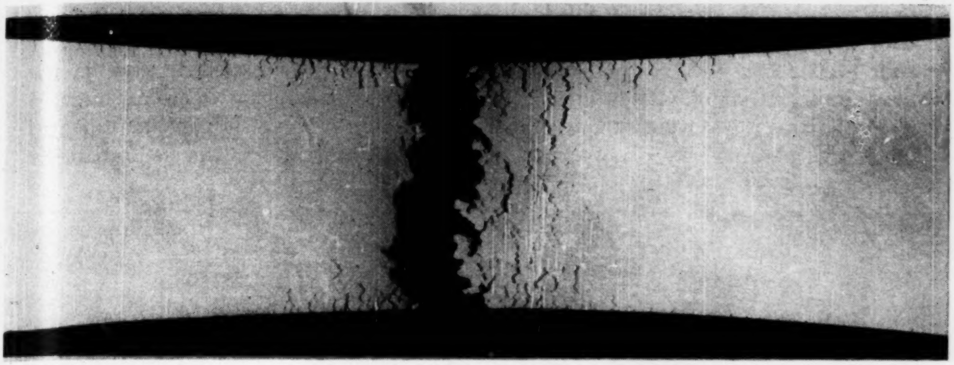
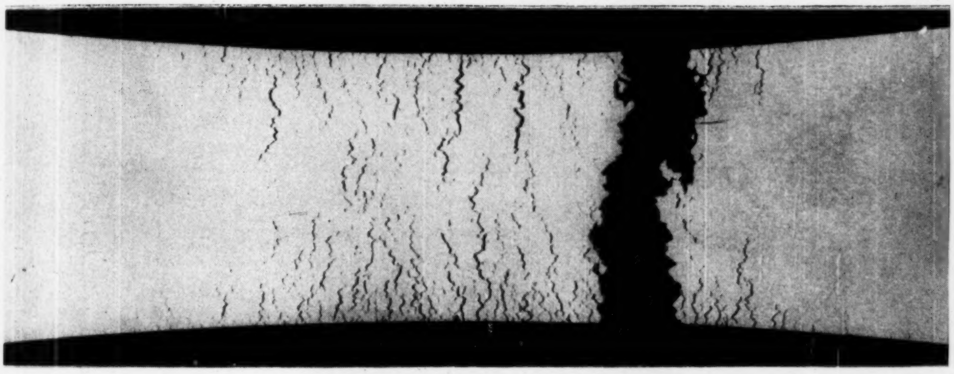


FIG. 2.—Creep-Rupture Results of Tapered Nickel Rods Tested in Air at 1500 F Showing the Anomalous Fracture Behavior.

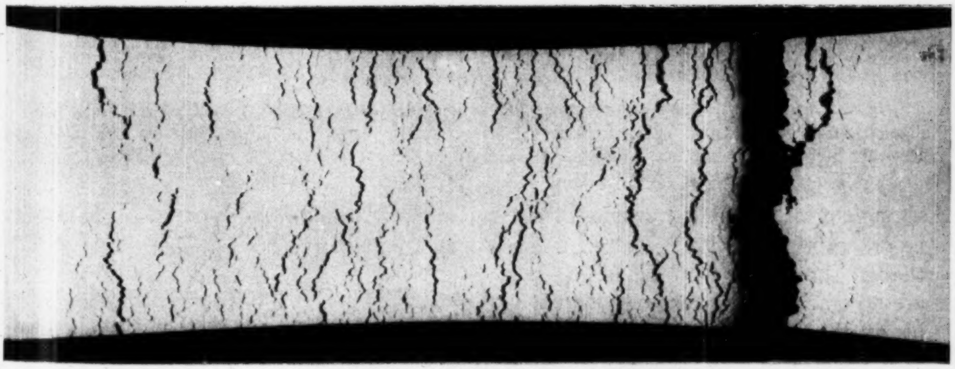
Star indicates interrupted, fractured by increasing load to 2360 psi.



(*a*) 4500 psi.



(*b*) 3000 psi.



(*c*) 2000 psi.

FIG. 3.—Macrographs of Unetched Longitudinal Center Sections of Tapered Nickel Specimens Creep Tested to Failure in Air at 1500 F (×9 before reproduction).

mum section were completely filled with oxide. All surface cracks were completely oxide-filled.

An appreciation of the sequence of events leading to the pattern described above was obtained by examination of specimens interrupted at a series of times before rupture. In Fig. 5 it can be seen that at the site of eventual failure there are no cracks at the center line after 300 hr but there are after 325 hr. It is shown quite clearly in this set of macrographs, as well as in Fig. 3, that cracking at the surface precedes that taking place internally.

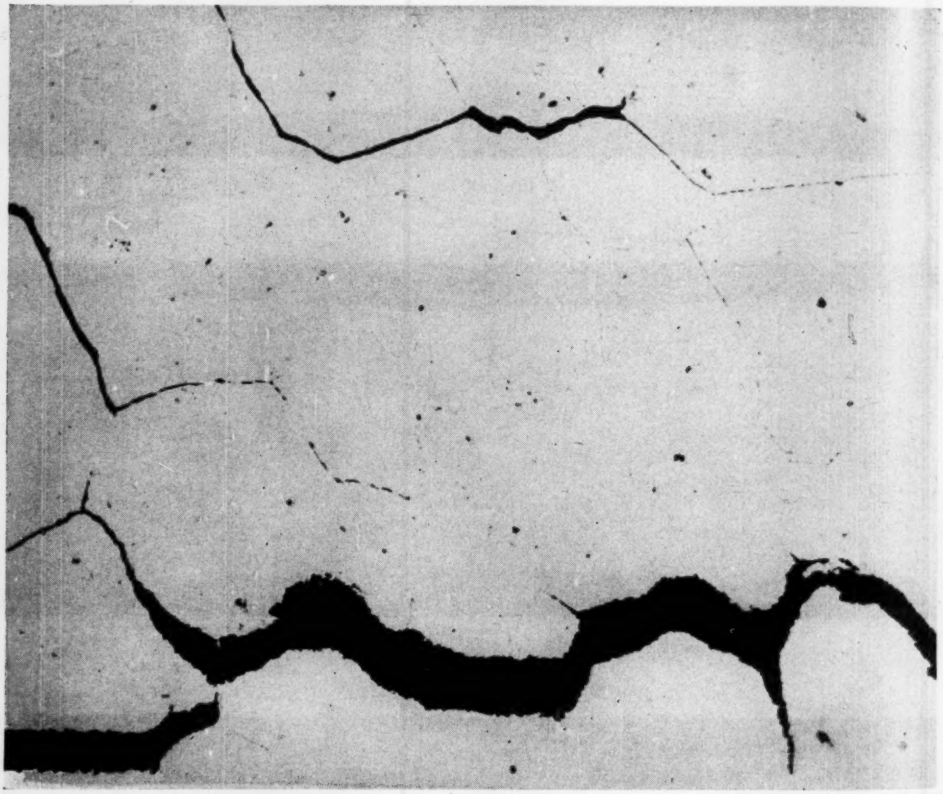
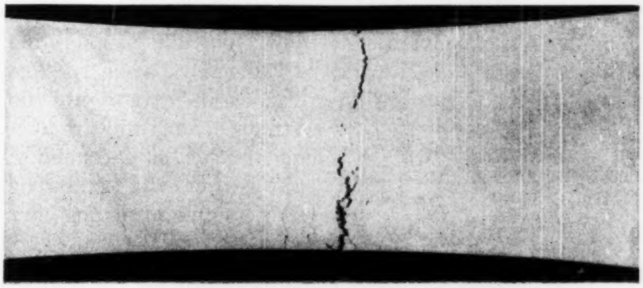


FIG. 4.—Micrograph Taken at the Incipient Fracture Showing Oxide-Free (*top*) and Fully Oxidized (*bottom*) Cracks in a Tapered Nickel Specimen Creep Tested in Air at 2000 psi and 1500 F. Rupture life, 350 hr; unetched (×250).

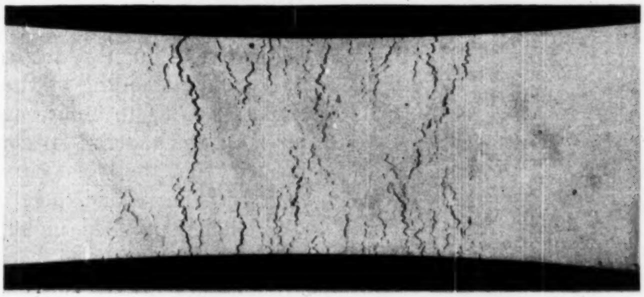
Microscopic examination of the sections in Fig. 5 reveals that all surface cracks are fully oxidized, even in the early stages. Cracks which are initiated internally, however, at first may be free of oxide, but those in the small-diameter sections become completely oxide-filled before fracture.

Conventional Rod-Type Specimens:

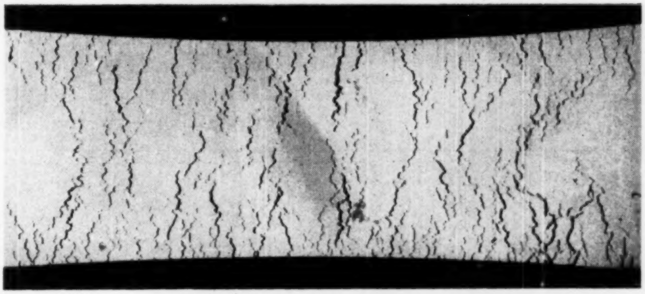
In the experiments described above it is difficult in tapered sections to distinguish between the effects of variations in section size and in stress. To supplement these data and make possible the separa-



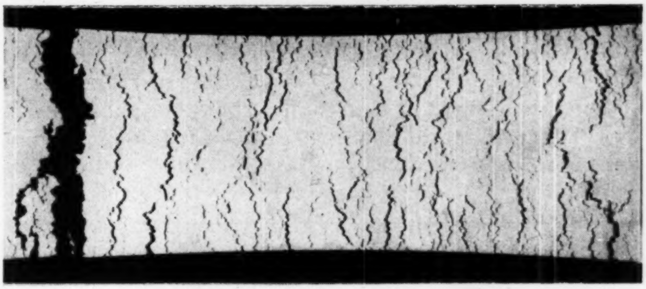
(*a*) Interrupted 263 hr.



(*b*) Interrupted 300 hr.



(*c*) Interrupted 325 hr.



(*d*) Fractured 350 hr.

FIG. 5.—Macrographs of Unetched Longitudinal Center Sections of Tapered Nickel Specimens Interrupted During Creep in Air at 2000 psi and 1500 F (×9 before reproduction).

tion of the two factors, creep-rupture tests were run on two sets of conventional specimens with diameters of 0.250 and 0.357 in. At each stress the rupture life was longer and minimum creep rate was lower for the smaller diameter section (Table II). The sharp change of slope in Fig. 6 indicative of pronounced oxidation strengthening appears at a higher stress for the smaller specimen. Accompanying the greater oxidation strengthening, however, is an embrittling effect; the elongation and reduction of area values are lower. The high values of elongation measured after long times is a consequence of the large volume of oxide generated in grain boundaries and is not indicative of ductility in the usual sense.

TABLE II.—CREEP-RUPTURE RESULTS OF CONVENTIONAL 0.250- AND 0.357-IN. DIAMETER SPECIMENS IN AIR AT 1500 F.

Stress, psi	Diameter, in.	Rupture Life, hr	Minimum Creep Rate, per cent per hr	Total Elongation, per cent	Reduction of Area, per cent
3000...	0.250	59.3	0.087	8.3	5.0
	0.357	47.0	0.106	10.7	5.8
2800...	0.250	76.8	0.053	9.1	4.4
	0.357	61.6	0.078	9.9	5.2
2700....	0.250	126	0.032	8.4	4.6
2600...	0.250	>2560[a]	...	17.7[a]	....
	0.357	74.6	0.055	8.7	5.4
2550....	0.357	123	0.029	9.2	3.5
2500....	0.357	>2460[a]	...	21.0[a]	...

[a] Interrupted, ruptured by adding shot to load pan; total elongation measured prior to addition of shot.

An understanding of the sequence of events leading to failure away from the neck of the tapered specimens may be gained from the creep curves of Fig. 7. Initially the second-stage creep rate of the smaller specimen, under the influence of the higher stress, is the greater. With the advent of intergranular cracking both go into third stage, but only the smaller survives to enter a fourth stage where the creep rate becomes lower than in the second stage. The interpretation given for this behavior is that the generation

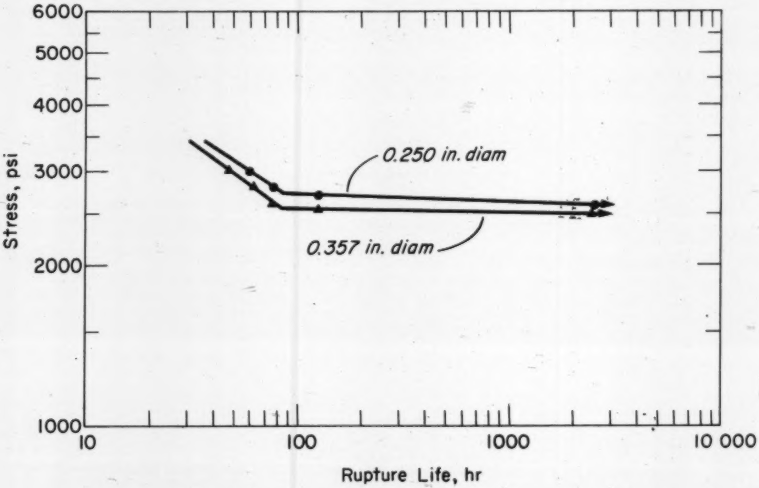


FIG. 6.—Initial-Stress Versus Rupture-Life Curves for Conventional 0.250- and 0.357-in. Diameter Specimens Creep Tested in Air at 1500 F.

of intergranular cracks results in accelerated transport of oxygen inward, with attendant oxidation strengthening of the interior. Since the distance oxygen must travel is shorter for the smaller section, it is more thoroughly strengthened. Consequently the seemingly anomalous result is observed that a longer rupture life results from a higher stress. In effect, it appears that the upper and lower curves of Fig. 7 represent the creep processes occurring in the small- and large-diameter section, respectively, of the tapered specimen.

A similarity also exists in cracking in the large- and small-diameter rods and the corresponding regions of the tapered ones. For example, after failure at 2800 psi there were more cracks in the interior of the smaller specimen than of the larger (Fig. 8). In the smaller section, detailed microscopic examination reveals that the internal cracks tend to be oxidized and appear to be continuations of cracks initiated at the surface, while in the larger section they are clean and more isolated (Fig. 9). Although the surface cracks of both size rods are oxide-filled, the proportion of the cross-section occupied by this oxidized layer is greater for the smaller diameter.

DISCUSSION

A mechanism for the displacement of fracture to a position of larger diameter is suggested by the creep results and metallographic observations. It is evident that some oxidation strengthening process acts to prevent or delay failure in small diameters. Several processes by which oxidation might increase rupture strength have been listed by Shahinian and Achter (2). In addition to the well-known process of hardening by internal oxidation, it was suggested that the solid oxide layers which bridge intergranular cracks can sustain a normal tensile stress and prevent fracture. In support of this

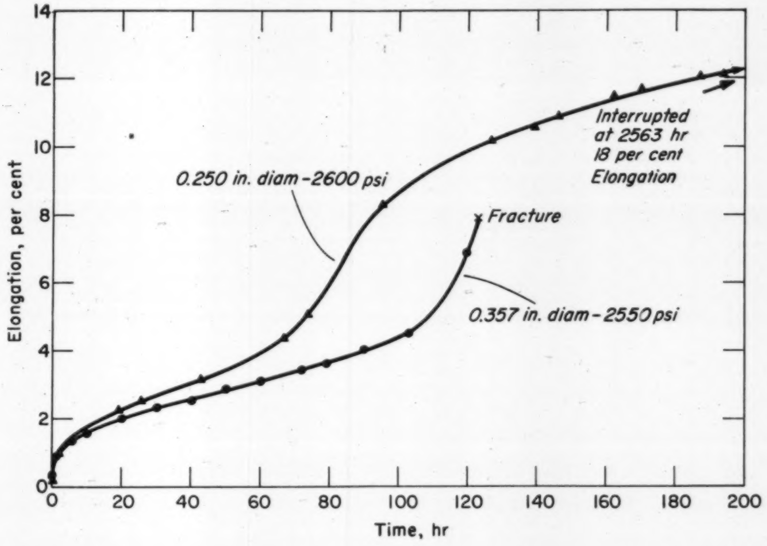
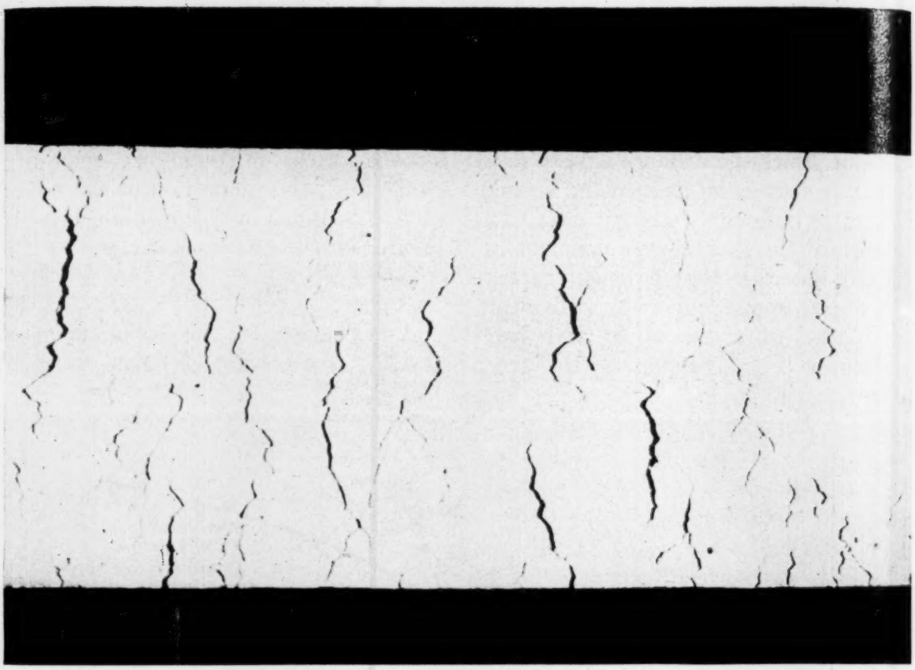
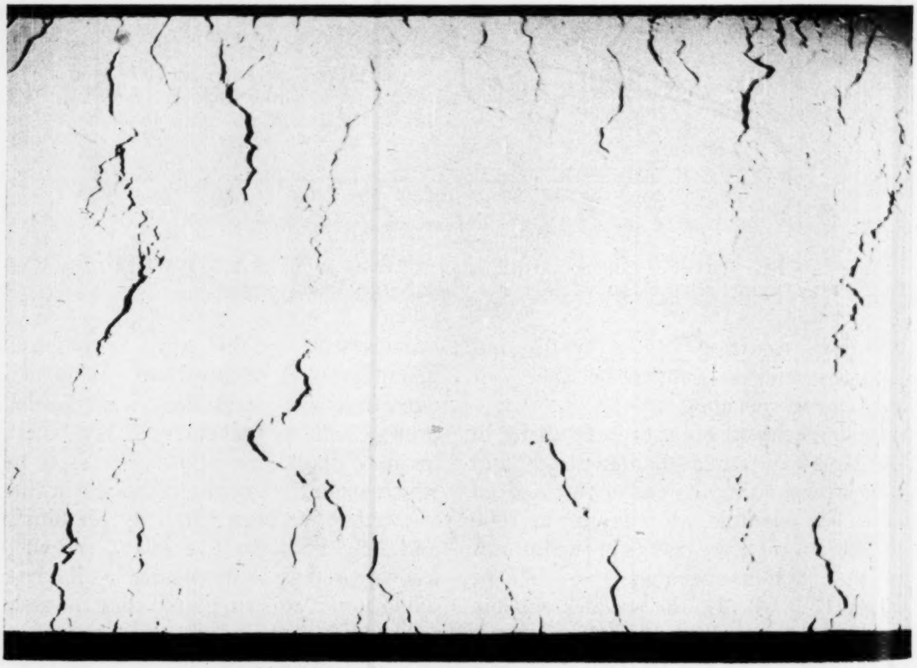


FIG. 7.—Creep Curves of Conventional Specimens Tested in Air at 1500 F Which Show That Failure May Occur Earlier Under a Lower Stress in a Larger Diameter Rod.



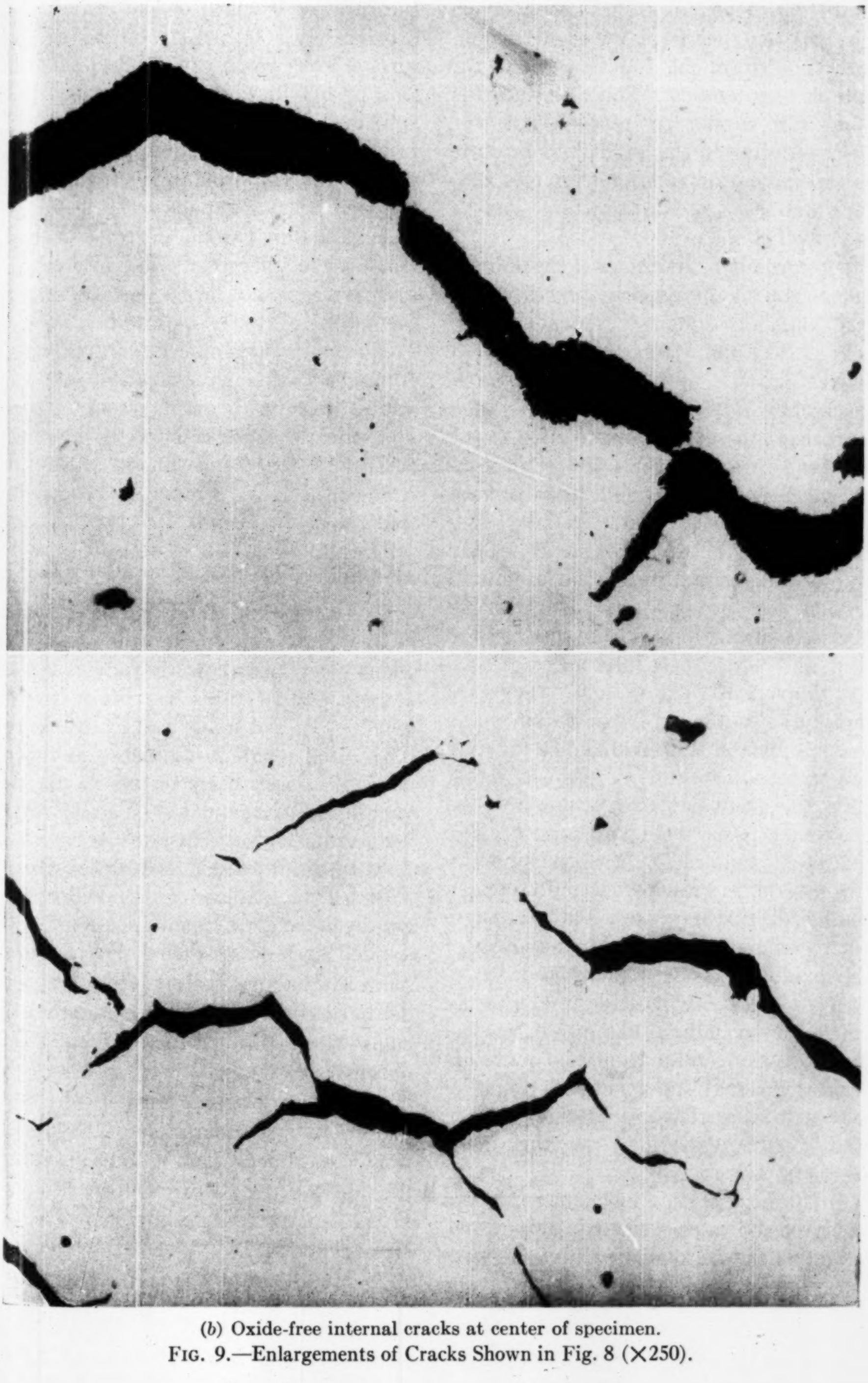
(*a*) 0.250-in. diameter rod after 76.8 hr (×10).



(*b*) 0.357-in. diameter rod after 61.6 hr (×10).

FIG. 8.—Crack Patterns in Longitudinal Sections of Rod Creep Tested to Failure in Air at 2800 psi and 1500 F.

(*a*) Fully oxidized crack at center of specimen.



(*b*) Oxide-free internal cracks at center of specimen.

FIG. 9.—Enlargements of Cracks Shown in Fig. 8 (×250).

mechanism it has been shown that the rupture strength of nickel oxide layers sintered together is of the right magnitude to perform this function (7). In the light of the demonstration that an oxide bond can support a tensile load, the events leading to the anomalous fracture of tapered nickel rods at high temperature and low stress (strain rate) can be described as follows:

Intergranular cracks first appear at the surface of the regions of small diameter and, subsequently, spread to the heavier sections. Later than the advent at the surface and at a slower rate, cracks also initiate and grow internally, starting at the neck and spreading to the heavier sections. In contrast to those at the surface, cracks in the interior are not always filled with oxide; initially they are clean but fill up subsequently. They do not cause fracture at the minimum section for two reasons. The heavily oxidized surface layers can support the load, and before these internal cracks can be propagated extensively, they are rapidly strengthened by oxidation made possible by the shorter distance for oxygen transport.

As the crack pattern extends into the heavier sections, two changes occur. The relative extent of surface cracking decreases and the transport path for oxygen to the interior increases. These changes act to reduce the strengthening effect of the oxidized surface cracks and to increase the extent of oxide-free internal cracks. Eventually a diameter is reached at which the combination of a more extensive internal crack network together with reduced surface oxidation lowers the load-bearing capability to the point where failure occurs.

In support of this mechanism, it can easily be shown that the oxidized surface layers at the neck of the taper can support the creep load. In Fig. 5(*b*) the surface crack depth at the neck is about 0.06 in. After a 300-hr sinter at 1500 F the rupture strength of the oxide is approximately 3000 psi (7). Therefore, the surface layer alone can support a tensile load of 111 lb whereas the applied load is 98 lb. It would appear, then, that rupture occurs at the section where the applied load is approximately equaled by the load-bearing capacity of the surface layer. For this reason a decrease in stress results in displacement of fracture position to a region of larger diameter where the oxidized layer is shallower.

The anomalous fillet fractures which stimulated this investigation are observed only in air at high temperatures and low strain rates. Since these are the conditions for displacement of rupture from the neck of the taper, it is concluded that the mechanism is the same for both.

SUMMARY AND CONCLUSIONS

The anomalous fillet fractures which nickel and nickel-base alloys exhibit when creep tested in air for long times at high temperatures have been investigated. Tapered rods and conventional 0.250- and 0.357-in. diameter specimens of nickel have been tested in air and vacuum at 1200 and 1500 F. The following results and conclusions are noted:

1. In air at 1500 F and stresses below 3500 psi, the tapered rods failed in progressively larger diameter sections as the applied stress was reduced. This displacement of fracture position from the neck of the taper occurred only under conditions where rupture life was greater in air than in vacuum.

2. Under the same air strengthening conditions, the smaller of the conventional specimens had a longer rupture life, indicating that the fracture behavior of the tapered rods results from size, not stress, effects.

3. Metallographic evidence shows that the greater strengthening of the smaller

sections is due to more complete bridging of intergranular cracks by load-bearing oxide. In the larger diameters, the relatively smaller penetration of oxidized surface cracks and the longer oxygen transport path to the interior permit the growth of oxide-free internal cracks which lead to failure.

4. It is concluded that the fillet failure of nickel in creep is due not to stress concentration but to preferential oxidation strengthening of the small-diameter gage section by load-bearing oxide.

Acknowledgment:

The aid of Mr. Joseph T. Atwell in the performance of the creep-rupture tests is gratefully acknowledged.

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(7) Unpublished research, U. S. Naval Research Laboratory.

DISCUSSION

MR. R. F. DECKER[1] (*presented in written form*).—The authors are to be congratulated on another in a series of papers which have thrown light on creep and atmosphere phenomena.

The degree of microcracking found during creep, especially that at 1500 F, is surprisingly high. Likewise, ductility is low. Do the authors consider that this is inherent from the deformation mechanism, or are there trace elements in the experimental nickel which enhance microcracking? Any chemical analyses that the authors could provide would be helpful in settling these questions.

MESSRS. T. C. REUTHER, P. SHAHINIAN, AND M. R. ACHTER (*authors*).—The only analysis we have available for the specific lot of nickel used for the tapered specimens is the supplier's "heat analysis." It is as follows: 0.03 per cent carbon, 0.01 per cent manganese, 0.007 per cent silicon, 0.01 per cent copper, 0.02 per cent iron and 99.8 per cent nickel. We have recently used 99.99 per cent nickel for some creep-rupture work and have found less intergranular cracking for the more pure material.

MESSRS. H. E. MCCOY AND J. R. WEIR[2] (*presented in written form*).—The phenomenon disclosed by the authors in this paper is quite interesting. However, there are several points relative to both the experimental work and the interpretation of the results that should be clarified.

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[2] Metallurgy Division, Oak Ridge National Laboratory, Operated by Union Carbide Nuclear Co., Oak Ridge, Tenn.

In examining the results shown in Fig. 2 of the paper, it is observed that the point marked with an asterisk is plotted at 1850 psi and approximately 2200 hr. The authors state that the specimen did not rupture until the load was increased to 2360 psi. What justification do the authors have for plotting this point on Fig. 2?

It is noted that actual stress rupture in one case was displaced from the minimum cross-section by approximately 0.25 in. The cross-sectional area at this point is 1.13 times the minimum area in the specimen. Using an activation energy for self-diffusion of 70 kcal per mole, a temperature increase of 5 C at the fracture position could account for the displacement in fracture position. For the other three specimens that showed a displacement in fracture position, a temperature increase of only 2 or 3 C could account for the observations. The authors should present evidence to invalidate this possibility.

The authors propose a mechanism that requires the formation of oxide-filled cracks wherein the oxide is continuous across the cracks and capable of supporting a load. The evidence presented in Figs. 4 and 9(*a*) of the paper indicates that the larger cracks are not filled with oxide that is continuous across the cracks. The authors mention some unpublished work in support of the proposed strengthening mechanism in which it was found that the oxide could support a 3000-psi load after a 300-hr sinter at 1500 F. It is difficult to determine the justification for using this in support of the mechanism since, as is shown in Fig. 5 of the paper, the maximum sintering time between the first appearance of surface cracks and rupture of the specimens is approximately 100 hr. The strengthening effect in this nickel could be due to internal oxidation of impurities and the resulting dispersion of oxide particles such as that observed by Grant et al.[3, 4]

MESSRS. REUTHER, SHAHINIAN, AND ACHTER.—The point marked with an asterisk in Fig. 2(*a*) was plotted at 1850 psi because that was the creep load for 2203 hr. At that time the load was increased in increments every half hour. Rupture occurred several minutes after the final load was applied. The value of 2360 psi was the stress at the point of failure—a position almost at the shoulder of the specimen. The point is plotted in Fig. 2(*b*) to show where the specimen was weakest after 2203 hr at 1850 psi in air.

In their second point Messrs. McCoy and Weir suggest that a temperature gradient of 3 to 5 C could cause the anomalous fracture shifts. If these gradients exist, this could indeed be true. However, there is no reason to assume that these gradients would exist only during tests in air at low stress and high temperature. If a temperature gradient caused the anomaly, it would also be observed in vacuum, in air at 1200 F and in air at 1500 F at the higher stress levels. Such is not the case.

In the calculation of the load supported by the oxide, we used the rupture strength measured by Cass and Achter at this laboratory. Nickel specimens were preoxidized at 1500 F, sintered together at the same temperature, and pulled to rupture. The rupture strength, after an initial steep rise, changes very little with sintering time so that the choice of 300 hr

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[3] R. Widmer and N. J. Grant, "The Role of Atmosphere in the Creep-Rupture Behavior of 80 Ni-20 Cr Alloys," *Transactions*, Metals Soc., Am. Inst. Mining, Metallurgical and Petroleum Engrs., Vol. 92, p. 885, Dec., 1960.

[4] A. S. Bufferd, K. M. Zwilsky, and N. J. Grant, "Hard Particle Strengthening of Refractory Metals for High Temperature Use Through Internal Oxidation," Final Report on Navy, Bureau of Aeronautics Contract NOas 59-6223-c, AD-245401, June, 1960.

for the sintering time is not critical. Finally, it is not necessary that the oxide be void-free to be able to support a load. Metallographic study of unruptured specimens after various times of sintering has shown that the interfaces in our sintered couples have a very similar appearance to the grain boundaries in our tapered creep specimens. They contain many voids which are not completely filled even after a 600-hr sinter.

The authors recognize that internal oxidation also contributes as a strengthening process in nickel and nickel-base alloys. Internal oxidation would penetrate equally along the tapered gage section. Therefore the strengthening contribution to a specimen would actually be greater in the large than in the small sections, for it would affect a greater volume (for a given linear penetration) in the larger sections. The authors therefore do not feel that the strengthening contributions of internal oxidation can account for the fracture anomaly.

MR. HOWARD S. AVERY.[5]—This paper involves a hypothesis that the oxide is actually stronger than the metal in carrying the load at high temperature. Some confirmation of this is perhaps contributed by our experience in creep testing a 26 per cent chromium - 20 per cent nickel alloy as part of a program to define the load-carrying ability of cast heat-resistant alloys over the range from 1800 to 2300 F.

These tests are made in furnaces heated with a platinum-rhodium alloy wire resistance winding. In one test intended to run for 10,000 hr, the furnace burned out after 7852 hr at 2200 F. The specimen was carrying the load of 300 psi satisfactorily at 2200 F, but it broke while it was being removed from the furnace. Examination of cross-sections showed widespread internal oxidation that penetrated to the center. The appearance of this dendritic and intergranular oxidation led to the conclusion that the oxide was at least as strong as the metal itself. Of course it was more brittle, which accounts for its fracture during handling.

We extrapolated the 2200-F time-to-rupture line on the customary log-log plot and plotted 7852 hr as though it were an ordinary rupture point. It appeared appreciably stronger than we would have guessed from extrapolation. This again suggests that our ceramic material, the oxide cementing the walls of what otherwise would be cracks, is as strong or stronger than the metal itself. This seems to add weight to the authors' hypothesis that nickel oxide is a strong material at these high temperatures.

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CREEP-RUPTURE AND FATIGUE PROPERTIES OF THREE NICKEL-BASE ALLOYS IN HELIUM ENVIRONMENT*

By F. J. Wall,[1] T. Hengstenberg,[2] and H. B. Gayley[1]

Synopsis

Creep-rupture and fatigue properties were obtained in helium environment for Inco 713C and Inconel X at 1300 F and for Inco 700 at 1500 F. The creep-rupture properties in helium were, for design purposes, equivalent to published results in air. Sufficient air fatigue data were not available to permit a comparison with the generated fatigue data in helium.

The use of helium as a working fluid in a closed cycle gas turbine is a departure from the usual oxidizing combustion gases. Therefore, information on materials in nonoxidizing environments was required to permit informed engineering design decisions with a minimum of uncertainty.

A review of the literature showed that some creep-rupture and fatigue testing in nonoxidizing environments had been done. The results of one study showed a marked lowering of short-time (up to 500 hr) creep-rupture and ductility properties of several commercial alloys (**1**).[3] Other studies indicated that the creep-rupture behavior of pure nickel and nickel-chromium alloys were altered in nonoxidizing environments (**2**–**4**). Preliminary fatigue test results at high amplitudes and very low cycling rates suggested a lowering of fatigue properties in vacuum at long times-to-rupture for fatigue (**5**).

The design of the closed cycle gas turbine rotating blades required a thorough knowledge of long-time metal behavior at operating temperatures. Therefore it was deemed necessary to perform long-time creep-rupture and fatigue tests in helium. Consideration of design requirements led to the selection of Inconel X and Inco 713C alloys for evaluation at 1300 F and Inco 700 alloy for evaluation at 1500 F in helium environment.

Test Materials

Inconel X and Inco 700 were supplied as fully heat-treated bar stock. Both alloys were prepared by induction melting in air.

Inco 713C was received and tested in the as-cast condition. Two methods of preparing the alloy were evaluated: air melting the master heat and casting in argon, and vacuum melting the master heat and casting in vacuum.

The chemical compositions and heat treatments are listed in Table I.

Specimen Preparation:

A combination notch - smooth bar specimen was used in all creep-rupture

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* Presented at the Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

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[2] Westinghouse Electric Corp., Central Laboratories, Materials Laboratories, Pittsburgh, Pa.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

TABLE 1.—CHEMICAL COMPOSITIONS AND HEAT TREATMENTS OF ALLOYS.

Alloy	Heat	Chemical Compositions, per cent													
		Nickel	Chromium	Iron	Molybdenum	Titanium	Aluminum	Manganese	Carbon	Silicon	Copper	Sulfur	Boron	Columbium	Zirconium
Inconel X	HT-8572X	72.48	15.09	6.78	...	2.67	0.95	0.74	0.07	0.29	0.04	0.007	...	0.86	...
	X-4183[a]	balance	11.20	1.30	4.9	0.43	5.8	0.016	0.13	0.10	0.05	0.004	...	2.25	...
Inco 713C	X-4168[b]	balance	11.20	0.97	4.6	0.60	6.0	0.040	0.12	0.20	...	...	...	1.80	...
	187[c]	balance	12.70	1.32	4.3	0.56	6.0	0.020	0.14	0.10	...	0.007	0.013	2.04	0.14
	XV-1637[d]	balance	12.90	1.00	4.4	0.69	6.0	0.010	0.12	0.05	...	...	...	1.83	...
Inco 700	HT-7652X	45.59	15.17	0.57	3.26	2.26	3.13	0.06	0.16	0.25	0.03	0.011	...	...	...

HEAT TREATMENT

Alloy	Solution Anneal			Aging Treatment		
	Temperature, deg Fahr	Time, hr	Quench	Temperature, deg Fahr	Time, hr	Quench
Inconel X	2100	4	air	1550 1350	20 20	air
Inco 700	2160	2	air	1600	24	air

[a] Creep-rupture tests: air melt, argon cast.

[b] Fatigue tests: air melt, argon cast.

[c] Creep-rupture tests: vacuum melt, vacuum cast.

[d] Fatigue tests: vacuum melt, vacuum cast.

tests (Fig. 1). With this type of specimen, an alloy notch-sensitive at a test temperature will generally exhibit notch failure prior to a smooth bar failure. The smooth bar portion is then available for additional testing. Because of the greater strength of Inco 713C, the test diameters of the Inco 713C specimens (0.310 in.) were made smaller than the Inconel X and Inco 700 specimens (0.357 in.).

The dimensions of the axial type fatigue specimen are shown in Fig. 2. The notched fatigue specimens were geometrically similar to the unnotched specimens with the exception of a V-notch having a minor diameter of 0.252 in., a major diameter of 0.360 in., and a radius of 0.007 in. located at the center.

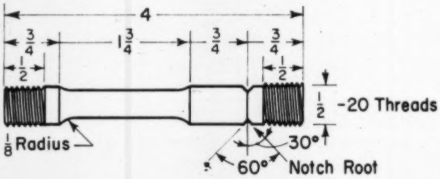
4
$\frac{3}{4}$
$1\frac{3}{4}$
$\frac{3}{4}$
$\frac{3}{4}$
$\frac{1}{2}$
$\frac{1}{2}$
$\frac{1}{2}$ -20 Threads
$\frac{1}{8}$ Radius
30°
60°
Notch Root

FIG. 1.—Combination Notch - Smooth Bar Creep-Rupture Specimen.

All dimensions in inches. Polish finish with No. 000 emery cloth.

Alloy	Major Diameter, in.	Minor Diameter, in.	Notch Root Radius, in.
Inconel X	0.500	0.357	0.010
Inco 700	0.500	0.357	0.010
Inco 713C	0.475	0.310	0.009

The configuration of the notch in both the creep-rupture and fatigue specimens was selected to give a theoretical notch concentration factor of 3.9 according to Peterson (6).

In view of the significance of cold working and residual stresses on fatigue and notch creep-rupture properties, considerable care was taken in preparing the specimens.

For the Inconel X and Inco 700 creep-rupture specimens, the notches were crush-formed ground to size. The Inco 713C specimen notches were machined to size with a single point tool ground to the notch contour.

The unnotched fatigue specimens were rough machined 0.025, in. oversize, rough ground to 0.008 in. oversize, then finish ground in 0.002 in. steps to 0.001 in. oversize. Polishing in turn with Nos. 180, 400, and 600 grit abrasives completed the preparation.

The Inconel X and Inco 700 notched fatigue specimens were rough machined to 0.020 in. oversize, and rough ground to 0.007 in. oversize. Following the finish griding operation, the notches were polished with a braided string using a 600-mesh abrasive. The notches of the Inco 713C specimens were rough machined to 0.010 in. oversize, and finish machined to size with a carbide tool ground to the

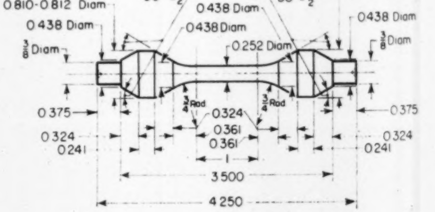
0810-0812 Diam
0438 Diam
$\frac{3}{8}$ Diam
30° $\pm\frac{1}{2}$'
0438 Diam
0438 Diam
0252 Diam
30° $\pm\frac{1}{2}$'
0810-0812 Diam
0438 Diam
$\frac{3}{8}$ Diam
0375
$\frac{3}{4}$ Rad
0324
$\frac{3}{4}$ Rad
0375
0324
0241
0361
0361
0324
0241
1
3500
4250

FIG. 2.—Axial Fatigue Specimen.

NOTE.—Finish to $\sqrt[8]{\text{surface}}$ quality all over (ASA B46.1). All dimensions in inches; all angles ± ½ min.

notch contour. Final polishing operation was the same as for the ground notch specimens.

Test System and Procedure

The basic creep-rupture test frames used for testing in helium environment were of the lever arm type having a mechanical advantage of 20 to 1. Temperature variation was held to ±3 F and thermocouples were attached to the lower, middle, and notched sections of the specimens. Bellows located on vacuum-tight furnace muffles permitted specimen elongations during the tests.

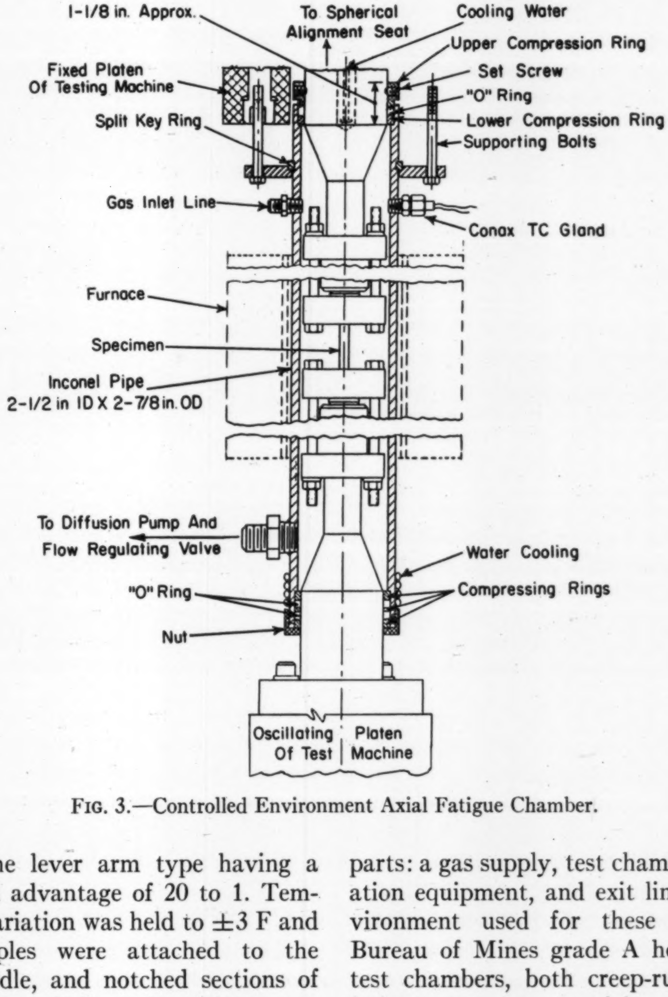
1-1/8 in. Approx.
To Spherical Alignment Seat
Cooling Water
Upper Compression Ring
Fixed Platen Of Testing Machine
Set Screw
"O" Ring
Split Key Ring
Lower Compression Ring
Supporting Bolts
Gas Inlet Line
Conax TC Gland
Furnace
Specimen
Inconel Pipe 2-1/2 in ID X 2-7/8 in. OD
To Diffusion Pump And Flow Regulating Valve
Water Cooling
"O" Ring
Compressing Rings
Nut
Oscillating Platen Of Test Machine

Fig. 3.—Controlled Environment Axial Fatigue Chamber.

For the fatigue tests in helium, vacuum-tight test chambers were designed and constructed for use on two axial type SF-4 fatigue machines (Fig. 3). In this machine, a specimen is subjected to a push-pull cyclic stress at a rate of 60 cps. Temperature variation is held to ±3 F and thermocouples are attached directly to the specimen.

The systems consisted of four main parts: a gas supply, test chamber, evacuation equipment, and exit line. The environment used for these tests was Bureau of Mines grade A helium. The test chambers, both creep-rupture and fatigue, were vacuum-tight muffles surrounded by standard resistance-wound furnaces. A 0.010 in. thick zirconium sheet lined the "hot zone" of the muffle and served as an oxygen getter. The evacuation equipment consisted of a mechanical fore pump, an oil diffusion pump, and appropriate pressure measuring devices. The pumps were capable of evacuating the systems to 1×10^{-2} μ at the test temperatures. The exit line consisted of a cold trap, surge trap, mercury column, and a flow rate measuring device. The cold trap prevented back diffusion of mercury vapor.

Prior to each test, the muffle, bellows, zirconium sheet, and specimen string were thoroughly cleaned and the system made leaktight. The system, back to and including the pressure gage on the gas container, was evacuated at a pressure level below 1 μ until a leak (decay) rate of 0.003 μ liters per sec was obtained. This procedure was repeated at about 400 F and again at test temperatures. At each temperature rise, the heating rate was sufficiently slow to ensure that the pressure in the system did not exceed 1 μ. For the fatigue tests, the specimen was cycled at 50 per cent of the test stress at the test temperature while the leak rate was measured. This was done to ensure that the vibratory loading did not produce leaks in the system.

TABLE II.—GAS ANALYSES FROM SEVERAL FATIGUE AND CREEP-RUPTURE TESTS.

Alloy	Test	Test Hours	Impurities, ppm									Remarks
			Acetone	Carbon Dioxide	Carbon Monoxide	Argon	Oxygen	Nitrogen Gas	Neon	Hydrogen	Total	
Inco 713C.........	fatigue.........	77	1.0	1.0	...	0.5	3.0	11.0	15.0	9.0	40.50	tank No. 105720
		348	0.1	0.9	0.6	0.1	0.3	3.5	14.0	13.0	32.23	tank No. 105720
Tank No. 105720...	...	...	...	0.2	...	0.2	1.8	6.0	10.5	0.5	19.20	
Inco 713C.........	fatigue.........	50	0.1	0.3	...	0.1	...	1.7	12.8	2.0	17.00	
Inco 700..........	fatigue.........	145	0.3	...	...	0.1	1.0	4.3	20.1	12.7	38.50	
Inconel X.........	fatigue.........	168	...	...	...	0.8	12.7	47.2	14.2	2.0	76.90	
Inco 713C.........	creep-rupture..	7020	0.2	0.3	...	0.1	3.6	11.3	13.2	2.6	20.20	
		7720	...	0.2	...	...	0.4	1.3	13.3	5.1	30.80	
Inco 700..........	creep-rupture..	700	1.3	0.8	...	...	2.3	6.4	13.7	28.7	53.20	
		500	2.9	1.0	...	1.3	5.0	29.5	13.1	21.6	74.40	
Inconel X.........	creep-rupture...	1914	3.1	0.3	...	0.7	10.0	18.1	13.9	43.3	89.40	

After the leak rate was obtained at the test temperature, helium was permitted to flow at a rate of $\frac{1}{4}$ cu cm per sec and at a pressure of 5 psig. The appropriate load was applied after the temperature of the specimen became stabilized.

A mass spectrometer technique was utilized to analyze impurities of low levels in helium (**7**). Essentially, the technique utilizes a cooling procedure to concentrate impurities to permit the analysis with a mass spectrometer. Several analyses are shown in Table II. Comparing impurity levels in samples taken from the systems during two tests with the impurity levels of a sample taken directly from the gas container (items 1, 2, and 3) shows that the total impurity levels were slightly higher in the test samples. However, the oxygen levels were similar. On other analyses, the total impurity levels are less than 100 ppm and the oxygen levels are less than 10 ppm in most cases. It should be noted that Bureau of Mines grade A helium generally contains a maximum of 50 ppm total impurities with less than 5 ppm oxygen.

Indications of the purity level of the

gas during a test can be realized by the relative discoloration of the specimen surfaces after testing. In this program, about one-half the creep-rupture and all the fatigue specimens were relatively bright. The balance of the creep-rupture specimens were slightly tinted.

TEST RESULTS

Prior to formal presentation of the data, several test criteria require explanation:

The fatigue strength is defined as the stress at which an alloy will accept 10^8 cycles. Combined load fatigue tests were made using an alternating-to-steady stress ratio of 0.25. The cycling rate used for the fatigue tests was 60 cps. At this rate 108,000,000 cycles represents 500 hr test time. The notch factor, K_f, is defined as the ratio of the unnotched-to-notched fatigue strength. The notch sensitivity factor, q, is defined as:

$$q = \frac{K_f - 1}{K_t - 1}$$

where K_t = theoretical notch concentration factor.

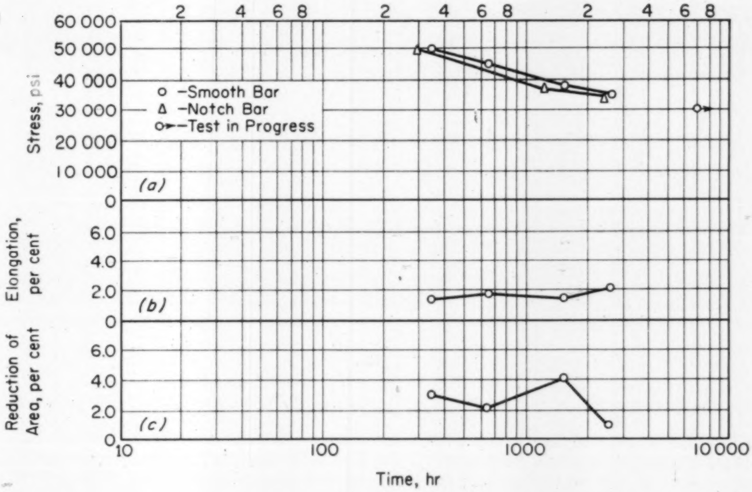

FIG. 4.—Creep-Rupture Properties of Inconel X at 1300 F in Helium.

Inconel X:

Creep-Rupture.—All but one of the Inconel X specimens tested in helium at 1300 F failed in the notch sections (Fig. 4(*a*)). However, there appears to be a general leveling off of both the notch and smooth bar curves at longer rupture times. This is substantiated by a test at 30,000 psi still in progress after 7000 hr. The estimated rupture time from published air data at 1300 F and 30,000 psi is 10,000 hr. Thus, it appears that the long-time rupture properties of Inconel X at 1300 F are not seriously lowered in helium.

The elongation values (Fig. 4(*b*)) measured on the ruptured smooth bar specimens were low, 1.5 to 2.0 per cent reflecting the marginal notch sensitivity of the alloy at 1300 F in helium. The reduction of area values (Fig. 4(*c*)) reached a minimum level at approximately 2500 hr.

Fatigue.—The stress-cycle data for Inconel X at 1300 F in helium are plotted in Fig. 5(*a*), and the fatigue strengths and notch factors are listed in Table III.

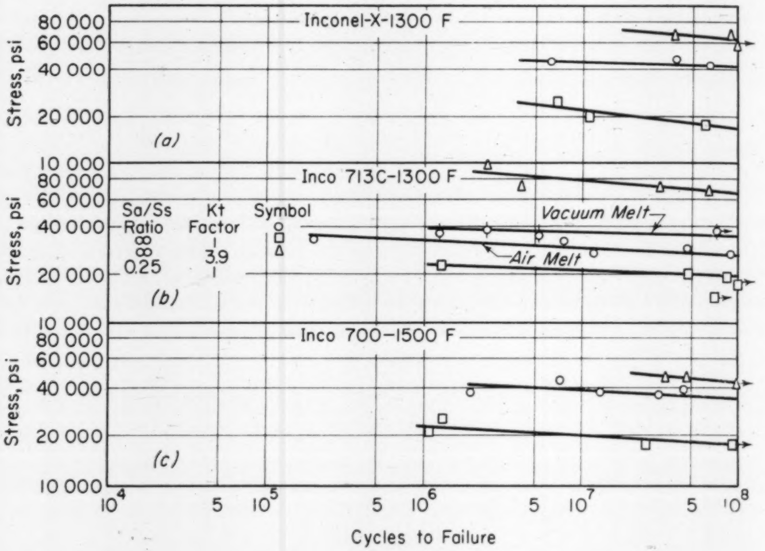

FIG. 5.—*S-N* Curves of Inconel X, Inco 713C, and Inco 700 in Helium.

TABLE III.—FATIGUE STRENGTHS AND NOTCH FACTORS.

Alloy	Temperature, deg Fahr	Atmosphere	S_a/S_s Ratio	K_t Factor	Fatigue Strength, psi	Notch Factor, K_f	Notch Sensitivity, q	Remarks
Inconel X....	1300	helium	...	1.0	41 000	...	...	
	1300	helium	...	3.9	17 000	2.4	0.48	
	1300	helium	0.25	1.0	62 000[a]	...	...	
Inco 713C....	1300	helium	...	1.0	26 000	...	...	air melt, argon cast
	1300	helium	...	3.9	19 000	1.4	0.14	air melt, argon cast
	1300	helium	0.25	1.0	64 000[a]	...	...	air melt, argon cast
	1300	helium	...	1.0	34 000	...	...	vacuum melt, vacuum cast
Inco 700.....	1500	helium	...	1.0	34 000	...	...	
	1500	helium	...	3.9	17 000	2.0	0.34	
	1500	helium	0.25	1.0	42 000[a]	...	...	

[a] Maximum stress.

The unnotched fatigue strength of Inconel X at 1300 F is 41,000 psi and the notched fatigue strength is 17,000 psi. The unnotched-to-notched fatigue strength ratio K_f is 2.4. The notch sensitivity factor, q, is 0.48.

The 500-hr combined load maximum stress ($a = 0.25$) was measured at 62,000 psi.

Stress-Range Diagram.—The 500-hr rupture stress on Inconel X at 1300 F in helium is 47,000 psi. The 500-hr fatigue stress is 41,000 psi and the 500-hr combined load cycle is 49,600 ± 12,400 psi. These points were used to construct the stress-range diagram shown in Fig. 6. As can be seen, the diagram is shaped as an ellipse.

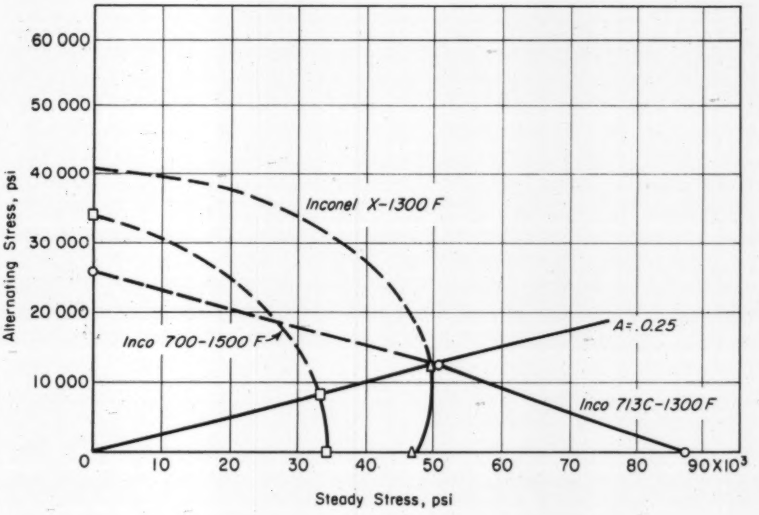

FIG. 6.—500-hr Stress-Range Diagrams—Helium Data.

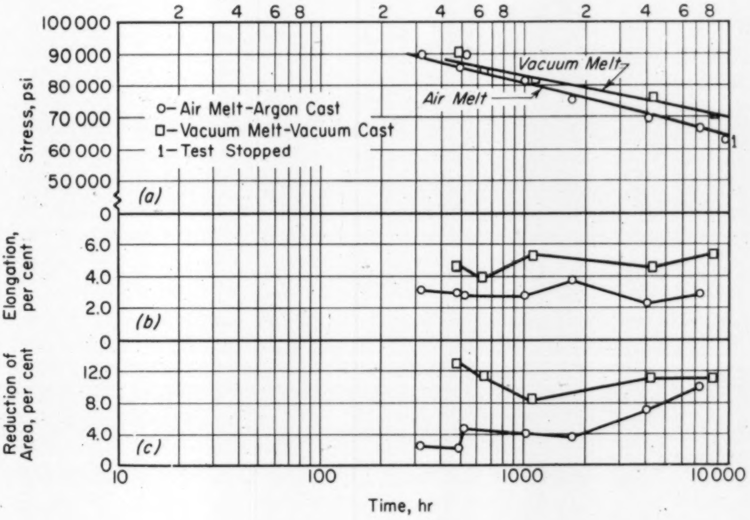

FIG. 7.—Creep-Rupture Properties of Inco 713C at 1300 F in Helium.

Inco 713C:

Creep-Rupture.—As shown in Fig. 7(*a*), the data obtained at 1300 F and high stress levels on air-melted Inco 713C alloy are somewhat scattered. For example, on two specimens tested at 90,000 psi, the rupture times were 320 and 520 hr; at 86,000 psi, the rupture time was 490 hr.

At high stress levels, the rupture properties for the vacuum-melted material are similar to those of the air-melted material. However, the long-time rupture properties of the vacuum-melted material are superior to those of the air-melted material. For example, the 10,000-hr rupture stress is about 70,000 psi for vacuum-melted material and 65,000 psi for air-melted material. These values are comparable to published air data.

As shown in Fig. 7(*b*), the elongation values measured on air-melted specimens are 2 to 3 per cent and relatively consistent over a wide range of rupture times—300 to 7000 hr. However, the elongation values measured on vacuum-melted material, 4 to 5 per cent, are distinctively higher than measured on the air-melted specimens over a similar range of time.

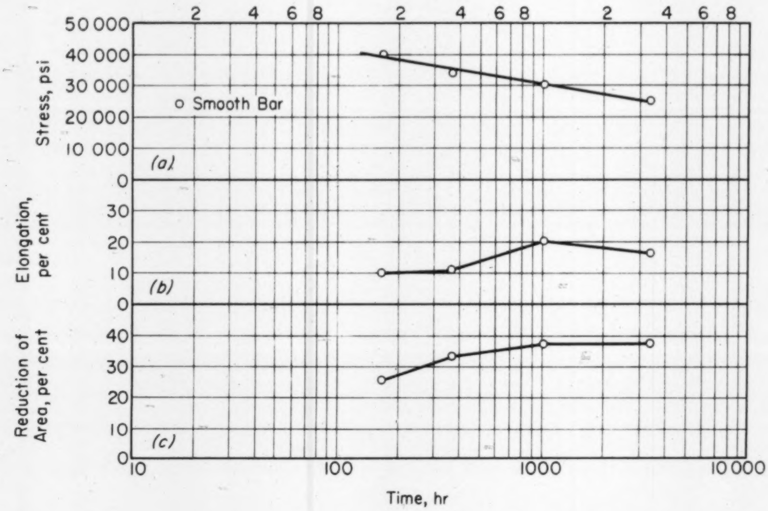

FIG. 8.—Creep-Rupture Properties of Inco 700 at 1500 F in Helium.

The reduction of area values measured on air-melted material increase with increasing rupture time (Fig. 7(*c*)). On vacuum-melted specimens, the reduction of area values range from 8 to 12 per cent and are higher than the values obtained on air-melted material.

It is interesting to note that even though the elongation values of the air- and vacuum-melted material are rather low at 1300 F, they are relatively consistent over a wide range of rupture times and do not yield a minimum value.

Fatigue.—The stress-cycle data obtained on Inco 713C at 1300 F in helium are plotted in Fig. 5(*b*). The fatigue information is listed in Table III.

For air-melted material, the unnotched fatigue strength at 1300 F is 26,000 psi and the notched fatigue strength is 19,000 psi. The unnotched-to-notched fatigue strength ratio K_f is 1.4 and the notch sensitivity factor q is 0.14. The notched fatigue characteristics of this alloy may be related to grain size, as coarse grain materials generally exhibit good notch fatigue properties.

The 500-hr combined load maximum stress ($a = 0.25$) is 64,000 psi for air-melted material.

The unnotched fatigue strength of 34,000 psi for vacuum-melted material is 24 per cent higher than the 26,000 psi value obtained on air-melted material.

Stress-Range Diagram.—At 1300 F, the 500-hr rupture stress for air-melted material in helium is 86,000 psi. The 500-hr fatigue strength is 26,000 psi and the

500-hr combined load cycle is 51,200 ± 12,800 psi. The plot of these points (Fig. 6) shows that the stress-range diagram for Inco 713C and 1300 F approximates a straight-line relationship. This condition may be associated with the rather low test temperature for this alloy.

Inco 700:

Creep-Rupture.—The data obtained on Inco 700 at 1500F in helium are plotted in Fig. 8(*a*). The helium data are similar to published air data. For example, the rupture time at 1500 F and 30,000 psi in air is estimated at 1000 hr. Under the same conditions a specimen ruptured at 1038 hr in helium.

The elongation values (Fig. 8(*b*)) ranged from 10 to 20 per cent over a wide range of rupture times, up to 3500 hr. Also, the reduction of area values (Fig. 8(*c*)) were high, ranging from 25 to 37 per cent. These ductility characteristics reflect the relative insensitivity of Inco 700 at 1500 F to notches.

Fatigue.—The stress-cycle data obtained on Inco 700 at 1500 F in helium are plotted in Fig. 5(*c*). The fatigue information is listed in Table III.

The unnotched and notched fatigue strengths of Inco 700 at 1500 F are 34,000 psi and 17,000 psi, respectively. With these values, the unnotched-to-notched fatigue strength ratio K_f is 2.0 and the notch sensitivity q is 0.34.

The 500-hr combined load maximum stress ($a = 0.25$) is 42,000 psi.

Stress-Range Diagram.—The 500-hr rupture stress of Inco 700 at 1500 F in helium is 34,000 psi. The 500-hr fatigue stress is 34,000 psi and the 500-hr combined load cycle is 33,600 psi ± 8400 psi. The plot of these data points (Fig. 6) shows a circular relationship.

DISCUSSION

Although an analysis of the various metallurgical factors affecting the behavior of these alloys under the test conditions or of the failure mechanism in a nonoxidizing atmosphere is beyond the scope of this paper, a few salient points indicated on the combined load diagram (Fig. 6) are worth noting.

It is significant that of the three alloys evaluated, all reacted differently to a small alternating stress when compared on the basis of their 500-hr rupture life at the temperatures considered in this study.

The range-of-stress diagram for Inconel X at 1300 F indicates that the new stress reaches a maximum at some intermediate stress ratio. For example, an alternating stress amplitude of 25 per cent of the reversed stress 500-hr fatigue strength increases the allowable steady stress by 5 per cent over the 500-hr static rupture strength.

The straight-line relationship of Inco 713C alloy at 1300 F indicates an adverse effect on the mean stress of even very small values of alternating stress amplitude. This is indicative of the superior rupture strength of coarse-grained material under static stress while the reverse seems to be true under cyclic loading.

In the case of Inco 700 alloy at 1500 F where the range-of-stress diagram indicates a circular relationship, the steady 500-hr mean stress is unaffected by alternating stress amplitudes up to approximately 10 per cent of the reversed stress 500-hr fatigue strength.

SUMMARY

Creep-rupture and fatigue data obtained in helium have been presented on three nickel-base alloys. The results show that Inconel X, Inco 700, and Inco 713C are capable of withstanding gas turbine design stresses in helium environment.

Acknowledgments:

The authors wish to thank the Atomic Energy Commission, the Maritime Administration, and General Dynamics Corp. for their cooperation and support of this work. The tests were made at the Westinghouse Steam Division High Temperature Laboratory, Lester, Pa.; the Westinghouse Materials Laboratories, Pittsburgh, Pa.; and the New England Materials Laboratory, Medford, Mass.

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CREEP-RUPTURE BEHAVIOR OF HASTELLOY-X

By Matthew J. Donachie, Jr.,[1] and Robert G. Shepheard[2]

A limited amount of creep-rupture data is available on Hastelloy-X in air. More extensive data in air are necessary, however, if the existence of an environmental effect, as suggested in the literature (**1–3**),[3] is to be studied. Therefore, prior to developing data on the behavior of Hastelloy-X in vacuum or inert environments, it was decided to perform tests to substantiate and expand the limited air data published in the literature (**4**). In order to make most effective use of these data, tests were aimed at establishing a parametric representation of the creep-rupture behavior of Hastelloy-X in air. The results of these tests are reported herein.

Procedure and Test Results

A total of 46 creep-rupture tests was carried out on solution-treated Hastelloy-X bar stock $\frac{1}{2}$ in. in diameter. All tests were performed on specimens from two lengths of bar from the same heat. These tests were carried out at temperatures from 1350 to 1800 F and at stress, σ, levels from 4000 to 60,000 psi. Minimum creep rate, MCR, was determined in addition to rupture life, RL, in the majority of the tests. The results are shown in Tables I and II. Sufficient tests were carried out to establish a plot of log σ versus log RL at 1350, 1500, 1650, and 1800 F from about 1 to 1000 hr in air, as shown in Fig. 1. This plot compares favorably with published data. However, at higher temperatures some deviations are noted. A plot of log σ versus log MCR was also constructed from the experimental results, as shown in Fig. 2. A composite plot of rupture life and minimum creep rate versus stress is shown in Fig. 3.

Appropriate constants for extrapolation parameters (Larson-Miller, Manson-Haferd) were determined from the rupture data. The constant C in the Larson-Miller (**5**) parameter, $P = T(C + \log t)$ = constant at constant stress, was found to be 16.0. The constants T_a and $\log t_a$ in the Manson-Haferd (**6**) parameter, $K = (T - T_a)/(\log t - \log t_a)$ = constant at constant stress, were found to be 1000 R and 10.30, respectively. The results of the parameter determinations are shown in Fig. 4(*a*) and (*b*) where master curves of stress versus the appropriate parameter are plotted.

Discussion

The results of the creep-rupture investigation of Hastelloy-X, when plotted as log σ versus log RL (Fig. 1) or log σ versus log MCR (Fig. 2), yield a series of straight lines at each temperature. The very small scatter of the experimental data about the mean straight lines indicates that testing procedures were satisfactory. No published data are available on log σ versus log MCR for this material. In addition, there are relatively few data available on log σ versus log RL. These data are published in reference (4). As noted previously, the experimental results are not in complete agreement with the published data. These differences may stem from composition effects, size effects, and quantity of data. In view of the small scatter of the experimental data about mean straight lines through the data points, it is felt that the present data are representative of the creep-rupture behavior of Hastelloy-X.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

TABLE I.—RUPTURE LIFE AS A FUNCTION OF STRESS AND TEMPERATURE.

Stress, psi	Rupture Life, hr, at									
	1350 F	1400 F	1450 F	1500 F	1550 F	1600 F	1650 F	1700 F	1750 F	1800 F
4 000	...	...	...	...	...	...	...	...	...	375.5
5 500	...	...	...	...	...	...	890.4	...	...	60.5
8 100	...	...	...	...	...	...	152.5	...	...	11.5
9 000	...	...	...	1420.6	...	195.0	76.8	26.0	8.15	5.60
10 100	...	...	...	806	...	...	...	...	...	...
12 000	...	...	...	249.7	102.4	51.5	15.9	5.20	...	1.43
15 000	...	...	275.9	100.2	32.2	9.90	4.20	1.50	...	0.40
18 200	839.1	...	100.3	33.5	12.2	4.40	1.85	...	...	...
20 000	559.8	195.4	61.7	18.1	5.95	2.20	1.30	...	...	...
23 300	228.6	...	...	8.90	...	...	0.45	...	...	...
30 000	62.5	...	...	1.40	...	...	...	...	...	...
40 000	10.8	...	...	...	...	...	...	...	...	...
50 000	2.00	...	...	...	...	...	...	...	...	...
60 000	0.60	...	...	...	...	...	...	...	...	...

It is interesting to note that all constant temperature lines on log σ versus log MCR or versus log RL plots are essentially parallel over the ranges of creep-rate and rupture life investigated. The published data for log σ versus log RL diverge with increasing rupture life. The present results suggest (see log σ versus log RL for 1800 F) that the long-time rupture strength of Hastelloy-X may be underestimated if previously published data are used. The two characteristic types of fracture, transgranular and intergranular, were observed in this study. However, no "break" corresponding to this transition was observed in the log σ versus log RL curve. The regions of existence of these types of fractures are indicated in Fig. 1. It was observed metallographically that there was some increase in the amount of carbides after long times at elevated temperatures. This had no apparent effect upon the slope of the log σ versus log RL plots.

No master curves are available in the literature for Hastelloy-X; therefore the development of a suitable master curve was considered an essential product of these tests. Three correlating parameters were investigated: the Larson-Miller and Manson-Haferd parameters as noted previously and the approximate parameter proposed by Clauss (7). This latter parameter (M = log RL + 0.010 T = constant at constant stress) is extremely simple to calculate and has been suggested by Clauss to be applicable to stress versus rupture life curves of the type shown in Fig. 1. The master curve prepared with this parameter exhibited large scatter about the mean straight line and is not considered valid. The Larson-Miller and Manson-Haferd parameters are apparently more accurate and useful. The master curves utilizing these latter parameters (Fig. 4) show reasonably low scatter about a mean line.

TABLE II.—MINIMUM CREEP RATE AS A FUNCTION OF STRESS AND TEMPERATURE.

Stress, psi	Minimum Creep Rate, in. per in. per hr, at								
	1350 F	1400 F	1450 F	1500 F	1550 F	1600 F	1650 F	1700 F	1800 F
4 000....	...	...	...	...	...	...	...	...	1.09×10^{-4}
5 500....	...	...	...	...	...	...	1.80×10^{-5}	...	5.20×10^{-4}
8 100....	...	...	...	...	...	...	...	...	6.39×10^{-3}
9 000....	...	...	...	7.63×10^{-6}	...	1.76×10^{-4}	5.92×10^{-4}	...	1.24×10^{-2}
10 100....	...	...	...	1.89×10^{-5}	...	...	...	...	...
12 000....	...	...	...	9.21×10^{-5}	4.46×10^{-4}	...	3.46×10^{-3}	...	9.60×10^{-2}
15 000....	...	...	1.28×10^{-4}	4.84×10^{-4}	...	1.30×10^{-2}	...	1.23×10^{-1}	...
18 200....	1.33×10^{-5}	...	5.72×10^{-4}	1.66×10^{-3}	...	3.83×10^{-2}	6.20×10^{-2}	...	...
20 000....	3.38×10^{-5}	2.12×10^{-4}	1.18×10^{-3}	2.70×10^{-3}	...	6.65×10^{-2}	...	...	...
23 300....	1.75×10^{-4}	...	...	8.25×10^{-3}	...	...	3.08×10^{-1}	...	...
40 000....	1.21×10^{-2}	...	...	...	...	...	...	...	...
50 000....	1.20×10^{-1}	...	...	...	...	...	...	...	...
60 000....	4.16×10^{-1}	...	...	...	...	...	...	...	...

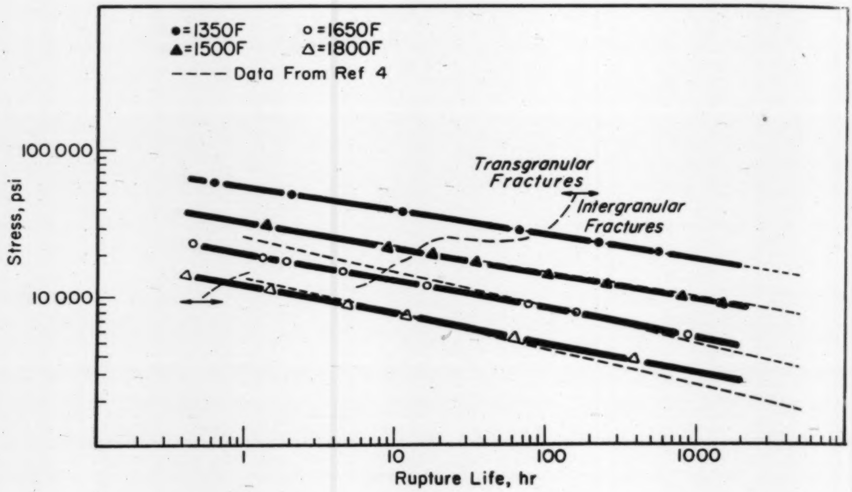


FIG. 1.—Stress Versus Rupture Life.

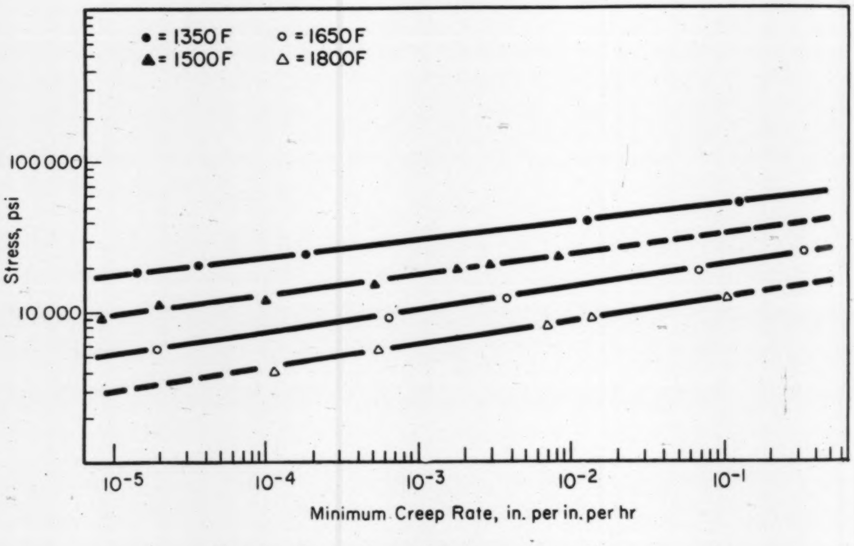


FIG. 2.—Stress Versus Minimum Creep Rate.

The predictions of all three parameters are compared in Table III for 1500 F and 10,000, 20,000, and 30,000 psi and against values taken from Fig. 1. The inapplicability of the approximate parameter is evident from the table, since at 10,000 psi it predicts a rupture life 700 hr longer than determined from Fig. 1. The Manson-Haferd parameter gave the best agreement with experiment over the entire stress range. This is in agreement with the general consensus of other

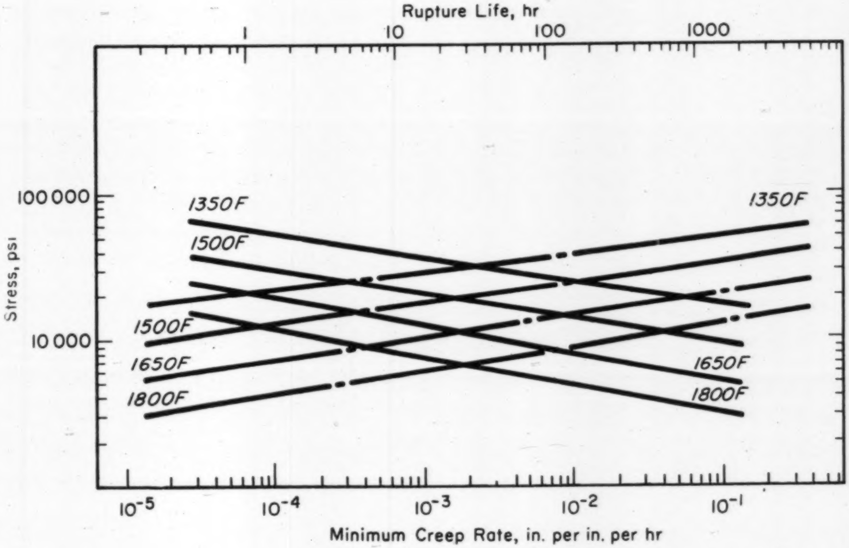
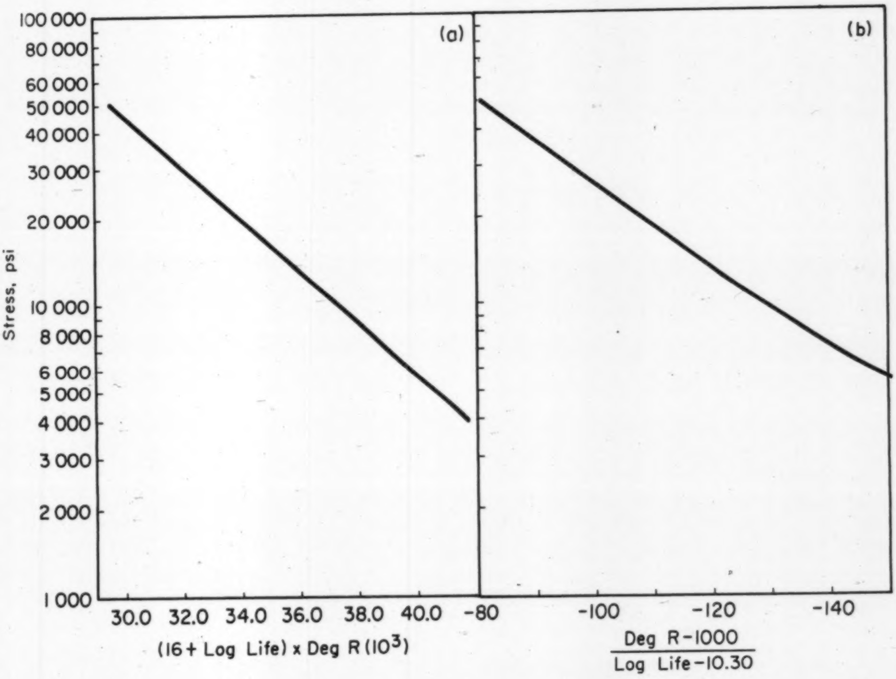


FIG. 3.—Stress Versus Rupture Life and Minimum Creep Rate.



(*a*) Using Larson-Miller parameter. (*b*) Using Manson-Haferd parameter.

FIG. 4.—Master Curves for Hastelloy-X Using Different Parameters.

TABLE III.—COMPARISON OF RUPTURE LIFE FROM FIG. 1 AND VALUES PREDICTED BY VARIOUS PARAMETERS AT 1500 F.

Stress, psi	Rupture Life, hr			
	From Fig. 1	Approximate Parameter	Larson-Miller Parameter	Manson-Haferd Parameter
10 000......	700	1413	1012	630
20 000......	20	20.0	20.9	23.4
30 000......	1.5	1.8	2.1	1.8

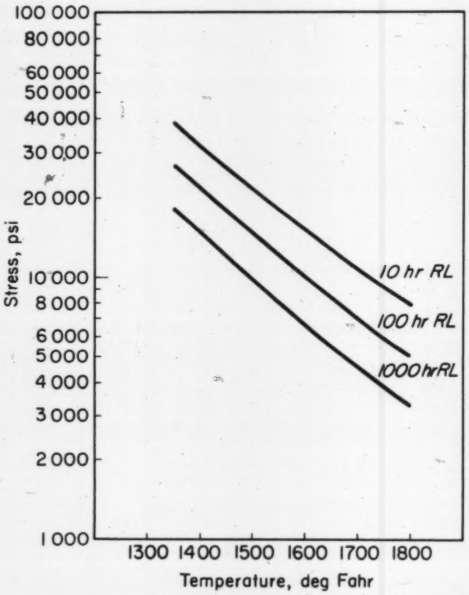


FIG. 5.—Stress Versus Temperature at Constant Rupture Life.

investigators. If a master curve is desired to extend the data to longer rupture times, then the master curve developed in this investigation using the Manson-Haferd parameter offers the most promise.

Since stress for a given rupture life is often desired, Fig. 5 gives a plot of stress for 10-, 100-, and 1000-hr rupture life versus temperature as derived from Fig. 1.

SUMMARY

The creep-rupture behavior of solution-treated Hastelloy-X bar stock has been investigated at temperatures from 1350 to 1800 F and stress levels from 4000 to 60,000 psi. The data obtained yield straight lines on a plot of log σ versus log RL and log σ versus log MCR and can be represented by a composite plot as shown in Fig. 3. The present experimental results differ somewhat at higher temperatures (1650 to 1800 F) from published data for this alloy in bar stock form. The long-time rupture strength of Hastelloy-X at the higher temperatures may be underestimated if previously published data are used.

Master curves using various parameters were constructed to correlate the rupture life data. The Manson-Haferd parameter with T_a = 1000 R and log t_a = 10.30 gave the best agreement with experiment over the temperature, stress, and rupture life range investigated.

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(2) P. Shahinian and M. R. Achter, "A Comparison of the Creep-Rupture Properties of Nickel in Air and in Vacuum," *Transactions*, Am. Inst. Mining, Metallurgical and Petroleum Engrs., Vol. 215, p. 37 (1959).

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(6) S. S. Manson and A. M. Haferd, "A Linear Time-Temperature Relation for Extrapolation of Creep and Stress-Rupture Data," *NACA Technical Note 2890*, March, 1953.

(7) F. Clauss, "An Examination of High-Temperature Stress-Rupture Correlating Parameters," *Proceedings*, Am. Soc. Testing Mats., Vol. 60, p. 905 (1960).

PROPERTIES OF HARDENED COPPER-BERYLLIUM STRIP AFTER EXPOSURES TO ELEVATED TEMPERATURES*

By K. G. Wikle[1] and N. P. Sarle[2]

Synopsis

This paper describes an investigation of the effects of exposing hardened (AT and HT) copper-beryllium alloy 25 (1.91 per cent beryllium, 0.26 per cent cobalt) at temperatures from 100 to 1150 F for 0 to 120 hr. Tensile and yield strength, proportional limit, elongation, and modulus values were determined. Results are compared with data reported on other copper-beryllium and copper-aluminum alloys and with the limited creep-rupture data available. Microstructural changes are illustrated and discussed.

Copper-beryllium alloy 25 (see ASTM Specification B 194[3]) in the age-hardened condition is recognized to be stronger, harder, and more fatigue-resistant than other commercially available wrought copper-base alloys. This strength is developed by a precipitation-hardening heat treatment at 600 to 750 F on material which is supplied in standard annealed or one-quarter, one-half, or full hard-rolled tempers. In spite of considerable experience and well-developed specifications concerning its room-temperature properties, very little has apparently been worked out defining the room- and elevated-temperature tensile properties of alloy 25 after short- and long-time exposures to elevated temperatures. A search of the literature (**1**, **2**, **3**)[4] shows that published work has been concerned mainly with binary copper-beryllium and ternary copper-cobalt-beryllium alloys containing from 0.2 to 0.5 per cent beryllium which exhibit only moderately high strength. On the other hand, alloy 25, the high-strength wrought copper-beryllium alloy, contains nominally 1.9 per cent beryllium. Thus, the spring, diaphragm, bellows, and fastener designers and users have had little information on engineering properties of this alloy by which to put it intelligently into applications where exposures to elevated temperatures are required. It was the purpose of this work to establish both the room- and elevated-temperature tensile properties of alloy 25 strip in the annealed-and-age-hardened (AT) temper as well as the hard-rolled-and-age-hardened (HT) temper after exposures up to 1150 F for

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[3] Specification for Copper-Beryllium Alloy Plate, Sheet, Strip, and Rolled Bar (B 194 – 55), 1958 Book of ASTM Standards, Part 2, p. 334.

[4] The boldface numbers in parentheses refer to the list of references appended to this paper.

periods as long as 100 hr. Short-time tension tests do not provide adequate information for extended use at elevated temperatures; however, they do define stress-time combinations where usage is definitely worth considering, rather questionable, or probably not worth considering. The data contained herein will define how exposures affect tensile properties and indicate temperature and time limits by which serious deterioration of such properties can be avoided.

TABLE I.—PROPERTIES OF AS-PROCESSED AND HARDENED STRIP.

Annealed:	
Tensile strength, psi	74 800
Elongation in 2 in., per cent	45
Rockwell hardness, 30 T scale	60
Average grain diameter, mm	0.020
Annealed-and-Age-Hardened (3 hr at 600 F):	
Tensile strength, psi	189 500
Elongation in 2 in., per cent	6
Rockwell hardness, 30 N scale	60
Hard-Rolled:	
Tensile strength, psi	112 500
Elongation in 2 in., per cent	7
Rockwell hardness, 30 T scale	82
Average grain diameter, mm	0.022
Hard-Rolled-and-Age-Hardened (2 hr at 600 F):	
Tensile strength, psi	206 000
Elongation in 2 in., per cent	3
Rockwell hardness, 30 N scale	62

TEST MATERIAL

Material tested in this program was selected from run-of-the-mill production. The rolling ingot was poured from a typical charge using a semi-continuous casting process described previously by Wikle (4). The charge was melted in a gas-fired furnace and cast into a 4 in. by 8 in. by 9 ft ingot. The ingot, after a homogenizing heat treatment to produce a uniform microstructure, was hot rolled to 0.250 in., bulk solution annealed at 1450 F, and cold rolled to 0.020 in. with two strand solution anneals at 1450 F, the first at 0.090 in. and the second at 0.032 in. The coil was then slit in half and one half was strand solution-annealed at 1450 F. Standard strip tension test specimens, 8 in. long with $\frac{1}{2}$ in. wide by 2 in. long gage sections, were prepared by slitting into $\frac{13}{16}$ in. wide strips and stamping out the bars on a punch press.

The chemical analysis of the heat was as follows: beryllium, 1.91 per cent; cobalt, 0.26 per cent; iron, 0.12 per cent; aluminum, 0.04 per cent; silicon, 0.10 per cent; nickel, 0.01 per cent; tin, 0.005 per cent; zinc, lead, and chromium, each <0.005 per cent.

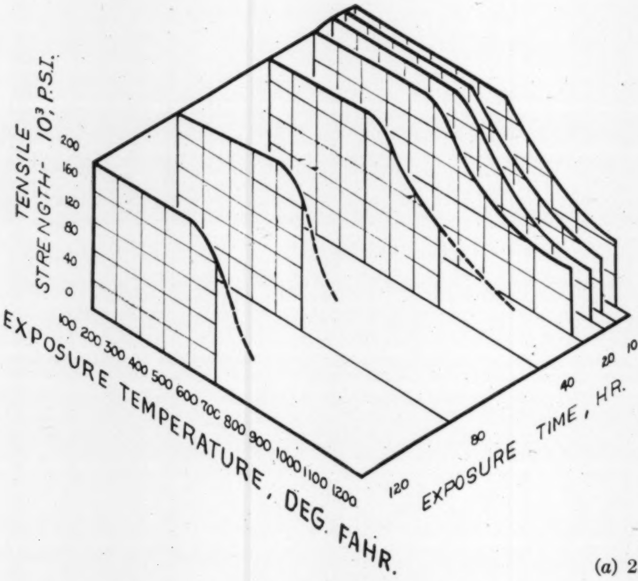
TABLE II.—EXPOSURE CONDITIONS.

Specimen Number	Temperature, deg Fahr	Exposure Times, hr
AT and HT bars for room-temperature evaluation:		
1	100	0-5-10-20-40-80-120
2	200	0-5-10-20-40-80-120
3	300	0-5-10-20-40-80-120
4	400	0-5-10-20-40-80-120
5	500	0-5-10-20-40-80-120
6	600	0-5-10-20-40-80-120
7	700	0-5-10-20-40
8	800	0-5-10-20-40
9	900	0-5-10-20
10	1000	0-5-10-20
11	1150	0-5-10-20
AT and HT bars for elevated-temperature evaluation:		
1	100	0-5-25-50-75-100
2	200	0-5-25-50-75-100
3	300	0-5-25-50-75-100
4	400	0-5-25-50-75-100
5	500	0-5-25-50-75-100
6	600	0-2-4-10-25-50-100
7	700	0-½-2-4-10-25-50-100
8	800	0-2-4-10-25-50-100
9	900	0-2-4-10-25-50-100
10	1000	0-2-4-10-25-50-100
11	1100	0-2-4-10-25-50-100

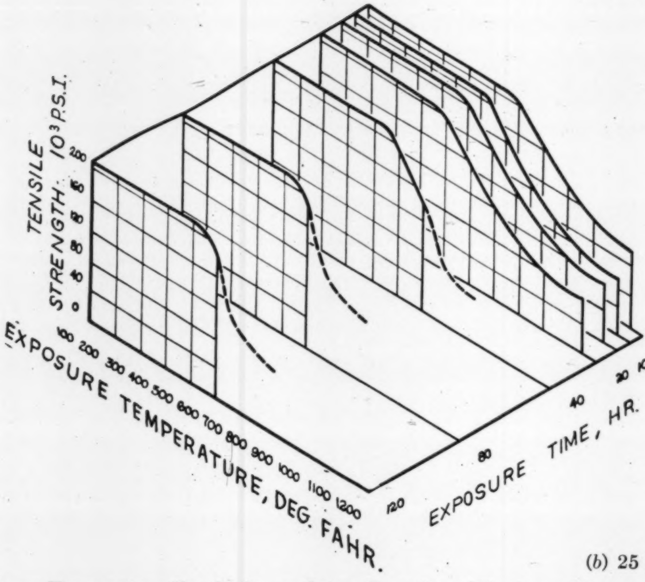
The as-mill-processed and precipitation-hardened tensile properties are as listed in Table I.

EXPOSURE CONDITIONS

After the preliminary heat treatment which put material of both tempers into



(*a*) 25 AT alloy strip.

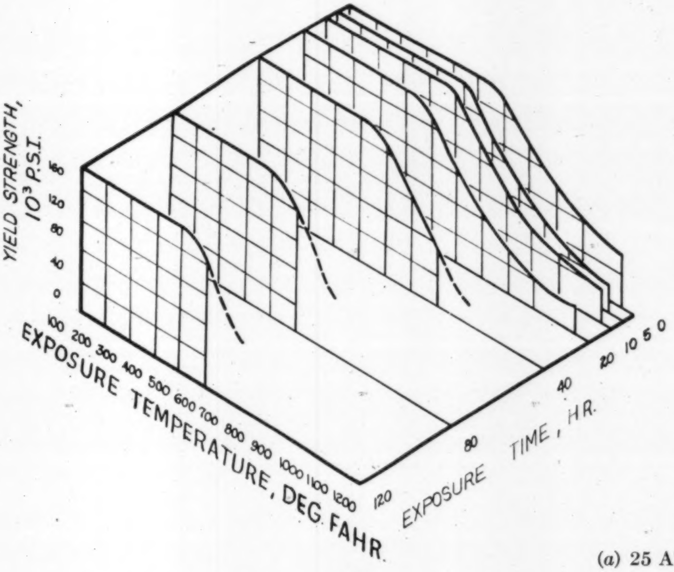


(*b*) 25 HT alloy strip.

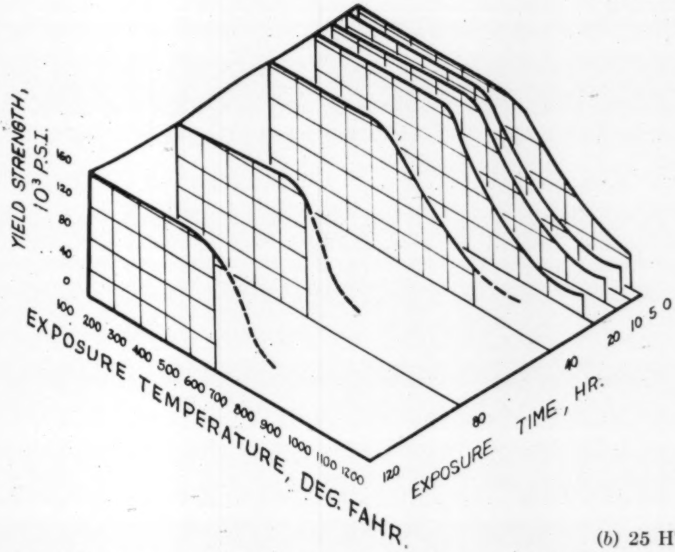
FIG. 1.—Room-Temperature Tensile Strength as a Function of Exposure Time and Temperature.

a precipitation-hardened state, the tension specimens were exposed to the combinations of time and temperatures given in Table II.

Specimens were loaded into a recirculating air furnace of relatively high heat capacity compared to the load, the furnace being set at the exposure temperature. Obviously, the introduction of the specimens somewhat suppressed the temperature of the furnace. However, the furnace regained its set temperature and brought the specimen temperature up to the set point in 10 to 15 min for



(a) 25 AT alloy strip.



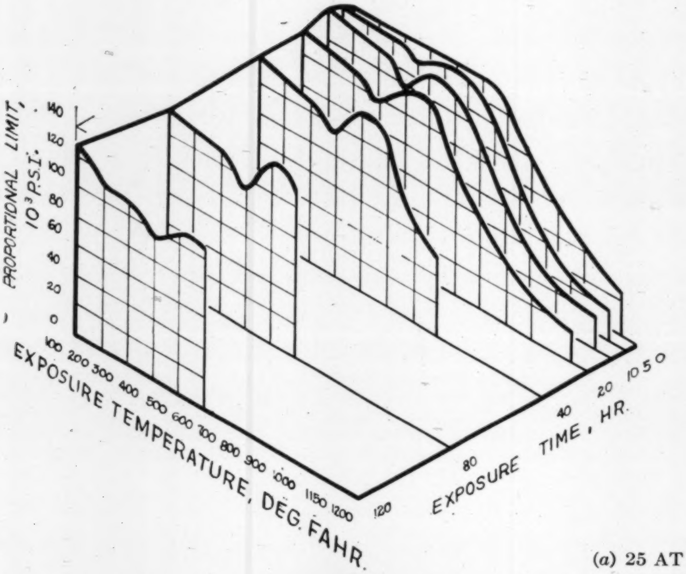
(b) 25 HT alloy strip.

Fig. 2.—Room-Temperature Tensile Yield Strength (0.2 per cent Offset) as a Function of Exposure Time and Temperature.

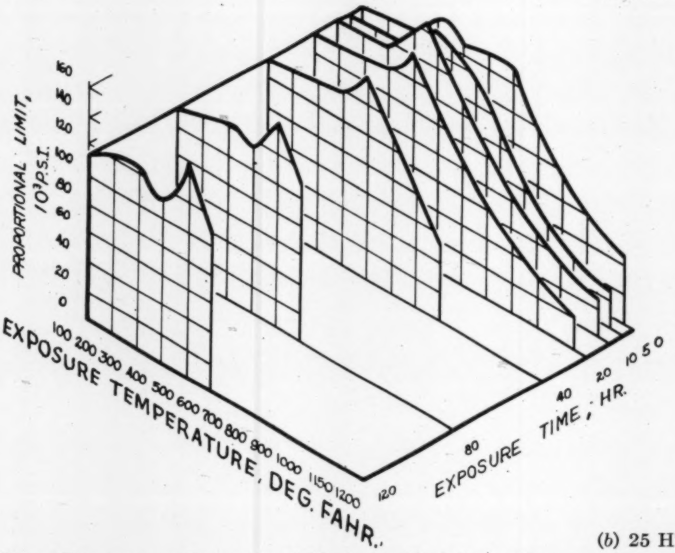
exposure temperatures of 600 to 1150 F. Below 600 F the rate of heating was predicted to have little effect and was not recorded. Exposure times started when thermocouples attached to the specimens indicated that the bars had just reached the exposure temperature.

Test Procedure

Tests at both room and elevated temperatures were conducted at a strain rate of 0.005 in. per in. per min. Elevated-temperature testing was carried out according to ASTM Recommended



(*a*) 25 AT alloy strip.



(*b*) 25 HT alloy strip.

FIG. 3.—Room-Temperature Proportional Limit as a Function of Exposure Time and Temperature.

Practice E 21[5] with the following exceptions: At 100 through 600 F the bars were heated to the test temperature in 35 to 40 min and then held at this temperature 15 min before testing. Above 600 F the specimens were pulled after holding at the test temperatures for a maximum of 5 min. This latter procedure

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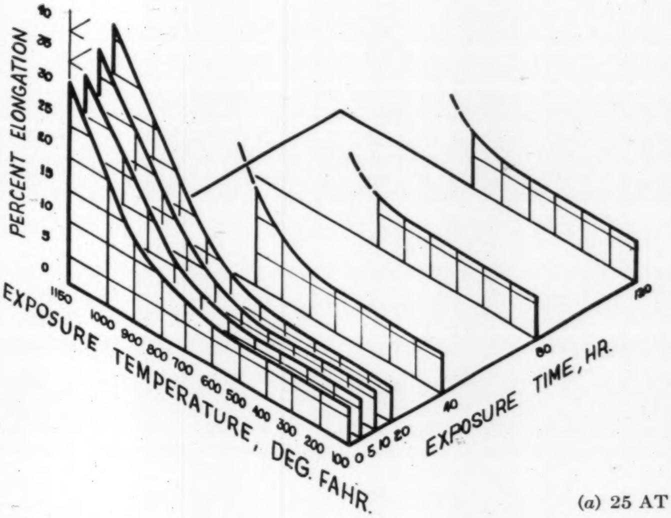
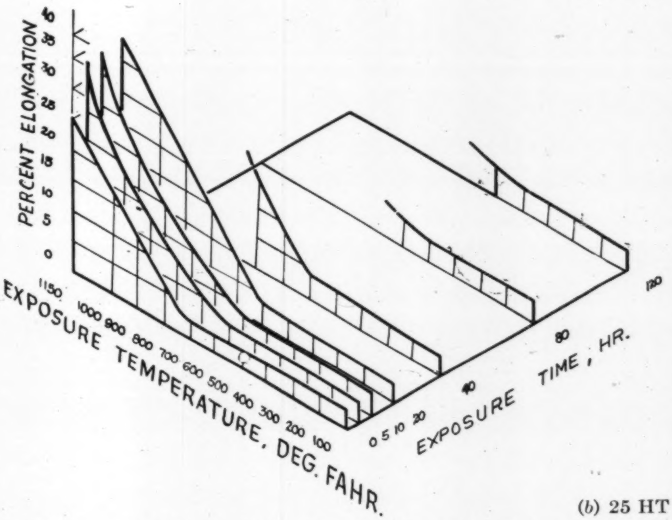
[5] Recommended Practice for Short-Time Elevated Temperature Tension Tests of Materials (E 21 - 58 T), 1958 Book of ASTM Standards, Part 3, p. 217.



(a) 25 AT alloy strip.



(b) 25 HT alloy strip.

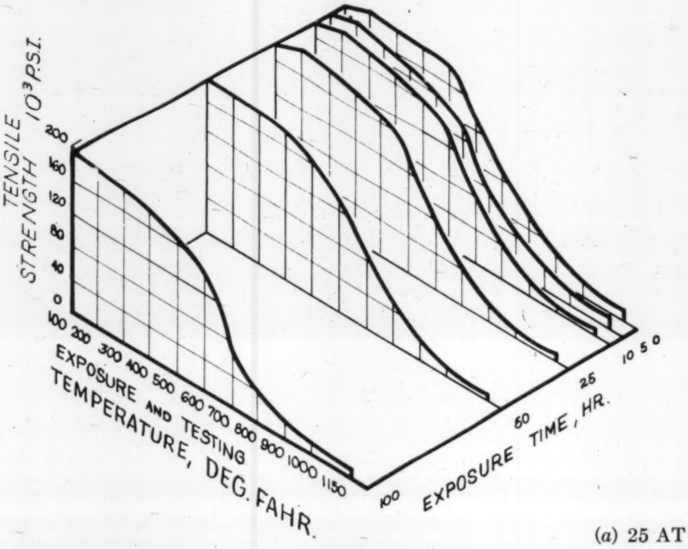
FIG. 4.—Room-Temperature Tensile Elongation (per cent in 2 in.) as a Function of Exposure Time and Temperature.

was followed in order to avoid as much overaging as possible during the test. Some of the bars were tested with the light film of oxide formed during the exposures, while others were superficially ground through No. 600 abrasive to remove completely the oxidation products before testing.

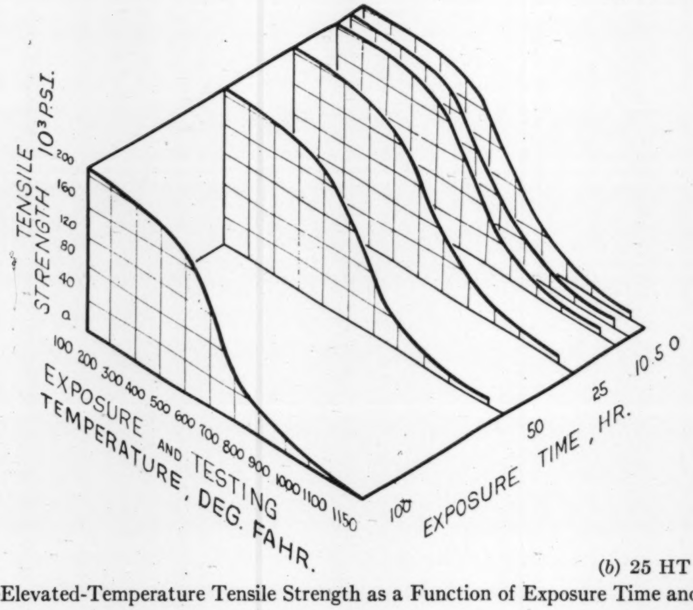
TEST DATA

Results of this evaluation program presented in three-dimensional property-time-temperature plots are as follows:

Room-temperature tensile strength in AT condition (Fig. 1(*a*)),
Room-temperature tensile strength in HT condition (Fig. 1(*b*)),



(*a*) 25 AT alloy strip.



(*b*) 25 HT alloy strip.

FIG. 5.—Elevated-Temperature Tensile Strength as a Function of Exposure Time and Temperature.

Room-temperature yield strength (0.2 per cent offset) in AT condition (Fig. 2(*a*)),

Room-temperature yield strength (0.2 per cent offset) in HT condition (Fig. 2(*b*)),

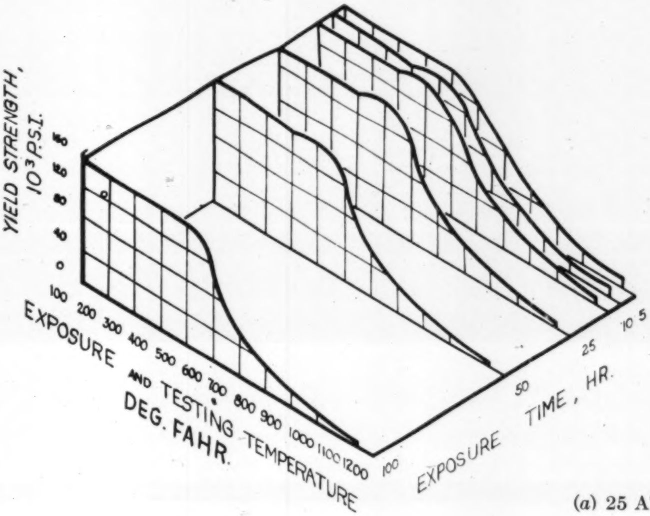
Room-temperature proportional limit in AT condition (Fig. 3(*a*)),

Room-temperature proportional limit in HT condition (Fig. 3(*b*)),

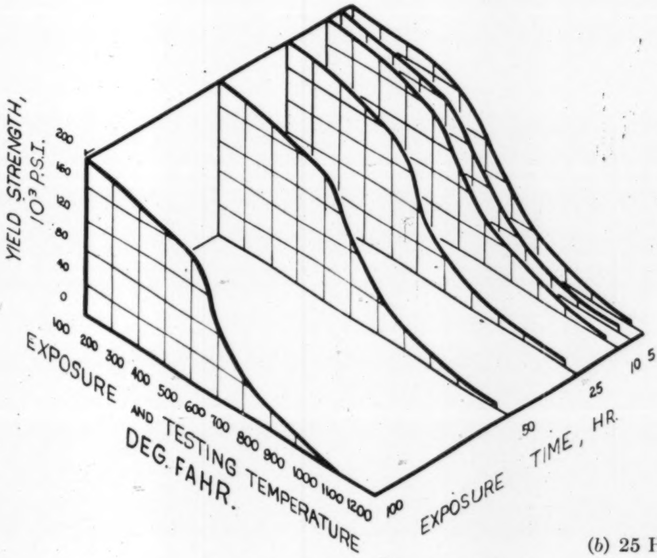
Room-temperature elongation in AT condition (Fig. 4(*a*)),

Room-temperature elongation in HT condition (Fig. 4(*b*)),

Elevated-temperature tensile strength in AT condition (Fig. 5(*a*)),



(*a*) 25 AT alloy strip.



(*b*) 25 HT alloy strip.

FIG. 6.—Elevated-Temperature Yield Strength (0.2 per cent Offset) as a Function of Exposure Time and Temperature.

Elevated-temperature tensile strength in HT condition (Fig. 5(*b*)),

Elevated-temperature yield strength (0.2 per cent offset) in AT condition (Fig. 6(*a*)),

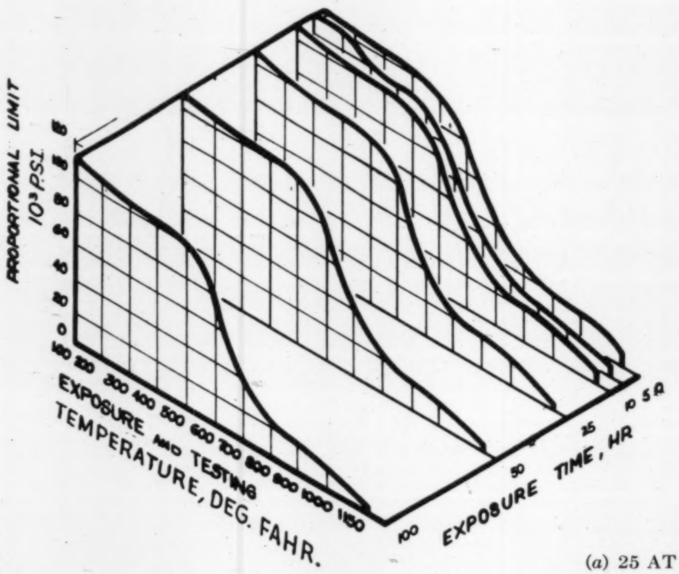
Elevated-temperature yield strength (0.2 per cent offset) in HT condition (Fig. 6(*b*)),

Elevated-temperature proportional limit in AT condition (Fig. 7(*a*)),

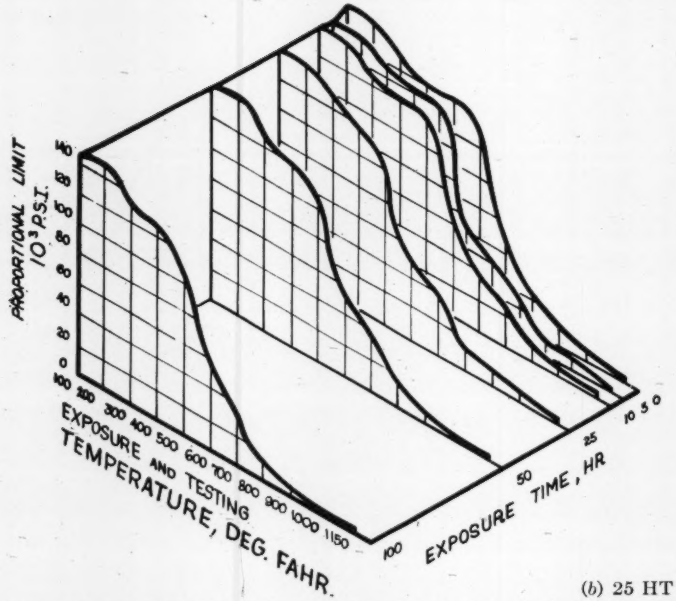
Elevated-temperature proportional limit in HT condition (Fig. 7(*b*)),

Elevated-temperature elongation in AT condition (Fig. 8(*a*)), and

Elevated-temperature elongation in HT condition (Fig. 8(*b*)).



(*a*) 25 AT alloy strip.

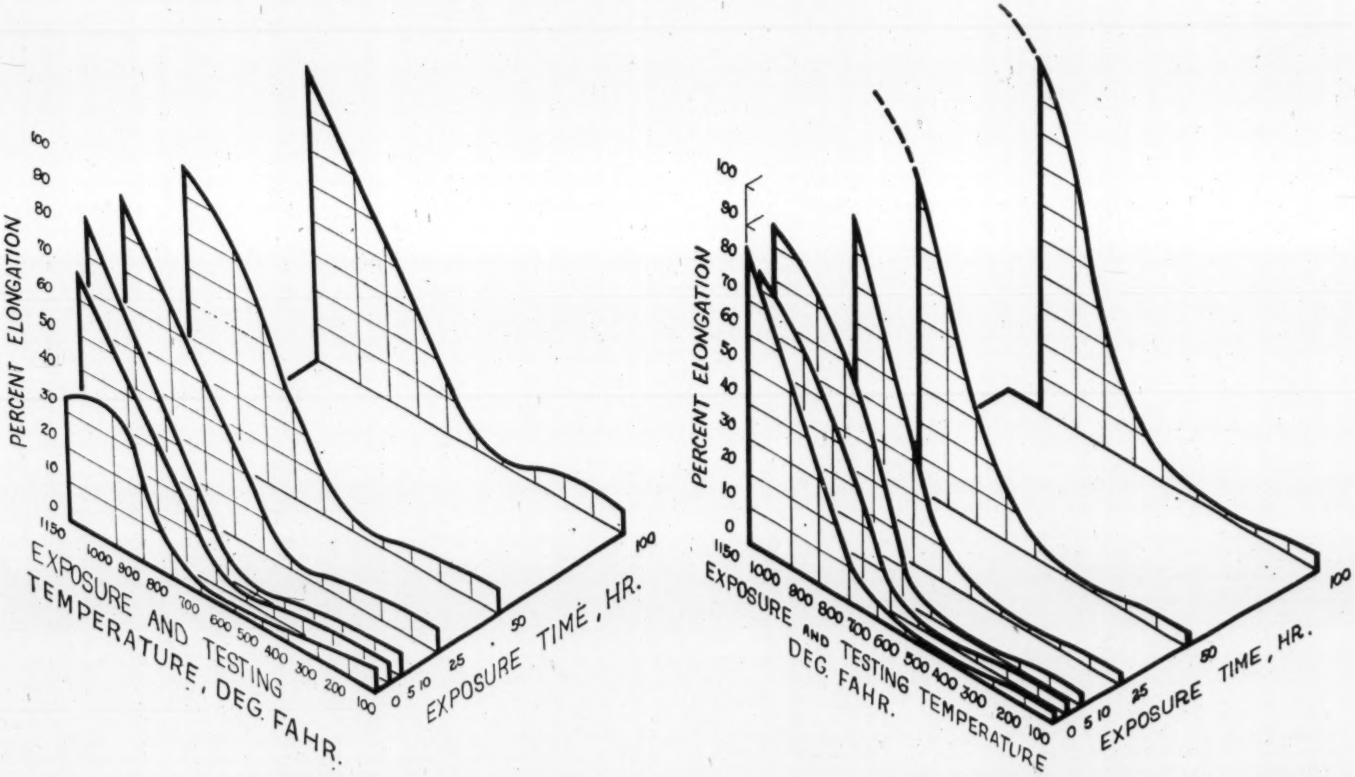


(*b*) 25 HT alloy strip.

FIG. 7.—Elevated-Temperature Proportional Limit as a Function of Exposure Time and Testing Temperature.

Also the modulus of elasticity of hardened alloy 25 in the AT and HT conditions at room temperature to 1150 F is plotted in Figs. 9(*a*) and (*b*).

No differences in properties were observed whether the test bars were tested "as-exposed" or after grinding off the thin oxide film to produce a clean speci-



(*a*) 25 AT alloy strip.

(*b*) 25 HT alloy strip.

FIG. 8.—Elevated-Temperature Tensile Elongation (per cent in 2 in.) as a Function of Exposure Time and Temperature.

men. Thus, results from both types of test specimens were averaged together to give final data values.

A study of the microstructural changes occurring in the 25-AT alloy strip from 0 to 100 hr while reheating from 600 to 1000 F is described in Table III and in Figs. 10(*a*) to (*h*). The structure was seen to change from (1) sharp grain boundaries, (2) coherent pre-precipitation and fine incoherent gamma precipitation in distorted slip planes (stress lines), and (3) light etching (in ammonium persulfate - ammonium hydroxide etchant) to a structure having (1) coarser precipitation, (2) diffuse grain boundaries and diffuse stress lines, and (3) dark etching. Finally, a dark-etching coarse "sorbitic" structure and then a light-etching "spheroidized" structure of large gamma globules in an alpha-copper matrix was reached at 1000 F for 0 and 100 hr, respectively. This would be expected of the gamma precipitation as overaging causes it to coalesce into coarse particles.

DISCUSSION OF RESULTS

Room-Temperature Properties:

The tensile strength and the yield strength of hardened alloy 25 in both the AT and HT conditions remained relatively constant for all exposure times studied up to 500 F. After 0-, 5-, and 10-hr exposure periods strength did not fall off until an exposure temperature of 700 F was exceeded. At the longer exposure ties there was a trend to further age harden up to 500 F but to soften markedly above this temperature.

The proportional limit, considered because of its close relationship to the elastic limit, decreased in the case of the AT condition as would be expected with higher exposure temperatures. However, this decrease was generally at a greater rate at longer exposure periods. A minimum was exhibited at 400 F and then a sharp increase occurred in exposing the bars at 500 F. Only a small decrease occurred after exposures at 600 F. This inflection probably represents where a stress-relieving mechanism which occurs by holding at 100 through 300 F is replaced by a precipitation-hardening mechanism which occurs in the range of 500 to 600 F. The latter temperatures represent the lower end of the normal precipitation-hardening range for alloy 25. The hardening trend noticed in the strength properties has a more significant effect on the proportional limit. In the case of the material in the HT condition, the proportional limit generally increased through exposures to 500 F. A decided dip occurred after 80- and 120-hr exposures at 400 F and a high peak was generally reached at 500 F. This demonstrates that long exposures at 400 F have a stress-relieving action, while long exposures at 500 F have a "finishing" effect on the precipitation-hardening phenomena.

Elongation is plotted in a reverse manner to the previous curves. This property remains relatively constant up to 600 F. Overaging at higher temperatures increases elongation. From these data, one could assume no "toughening" of hardened strip would occur until temperatures of 700 F and over were reached.

In Table IV the per cent change occurring during the maximum exposure period is listed. Here it is clearly indicated that only minor effects occur while heating both annealed and hardened as well as hard-rolled-and-hardened strip to 500 F for up to 120 hr. Such thermal treatment is generally beneficial to HT material and is beneficial at 500 F to AT material. This suggests age hardening

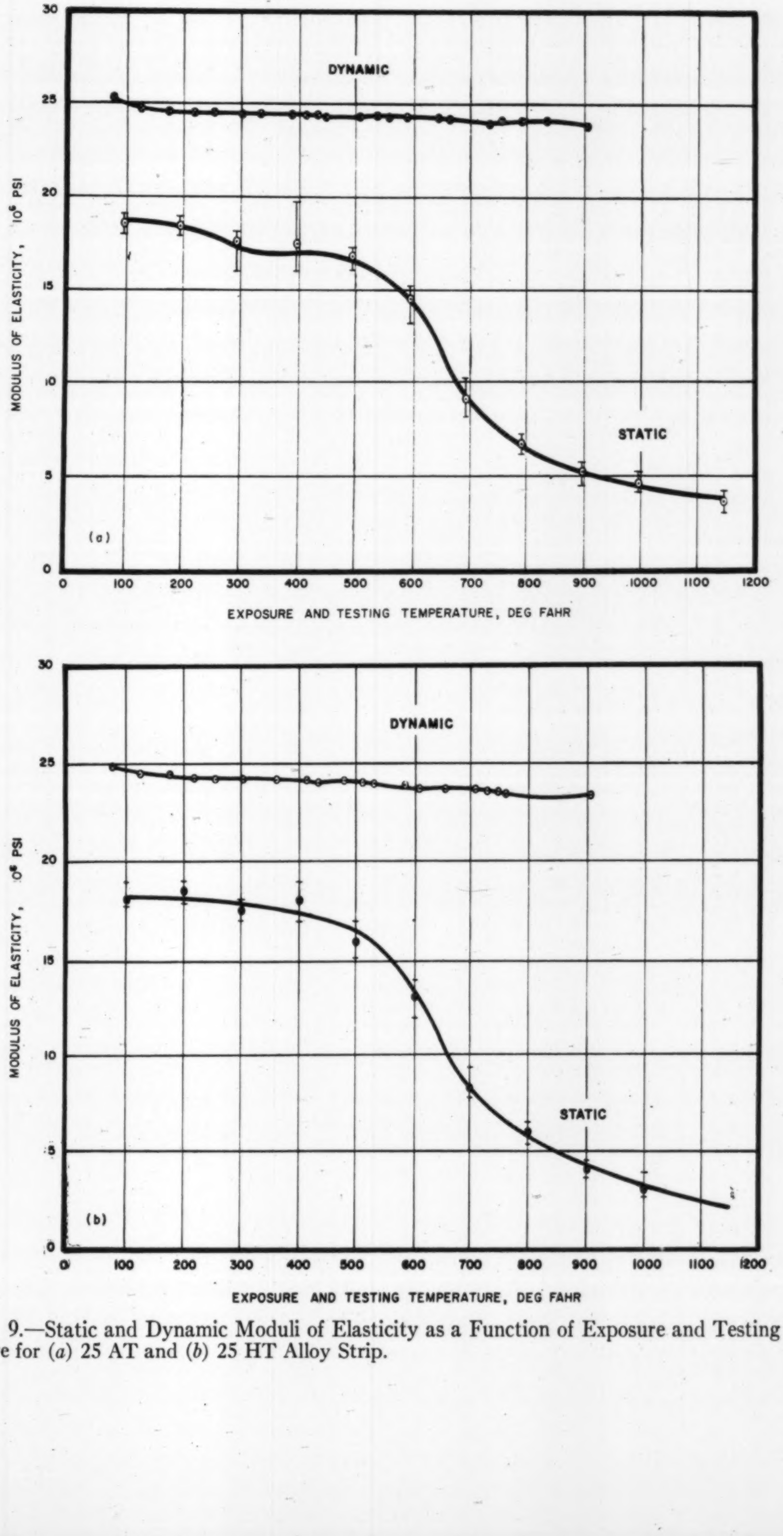


FIG. 9.—Static and Dynamic Moduli of Elasticity as a Function of Exposure and Testing Temperature for (*a*) 25 AT and (*b*) 25 HT Alloy Strip.

has resumed to take the material to a higher plateau. Such an effect suggests a potential advantage in a double aging treatment as is used in some other precipitation-hardening alloys.

TABLE III.—CHANGE IN MICROSTRUCTURE OF HARDENED (AT) 25 ALLOY STRIP REHEATED FROM ROOM TEMPERATURE TO 1000 F FOR UP TO 100 HR (AMMONIUM PERSULFATE - AMMONIUM HYDROXIDE ETCHANT).

Reheat Temperature, deg Fahr	Microstructure After Exposure to:	
	0 hr	100 hr
100 to 500.....	No significant change.	No significant change.
600.........	1. Clear structure of fine precipitation and distortion in slip planes (stress lines). 2. Grain boundaries sharp. 3. Some globular gamma precipitation in grain boundaries.	1. Not much change from 600 F, 0 hr. However, less distinct structure indicates more advanced precipitation.
700.........	1. Intragranular areas: darker etching, stress lines visible but less distinct. 2. Grain boundaries: less distinct, broader light etching zone of gamma precipitation.	1. Intragranular areas: dark etching, more diminution of stress line clarity. 2. Grain boundaries: broader coarser area of light etching gamma precipitation.
800.........	1. Intragranular areas: dark etching, stress lines diffuse. 2. Grain boundaries: less sharp, broader light etching zone of coarser gamma precipitation.	1. Intragranular areas: dark etching granular appearance, stress lines very diffuse. 2. Grain boundaries: very broad zone of coarse gamma precipitation. No boundaries visible.
900.........	1. Intragranular areas: dark etching, coarser gamma precipitation. Stress lines diffuse. 2. Grain boundaries: hard to distinguish. Broad zone of coarse, light etching gamma	1. Intragranular zone: coarse sorbitic structure. 2. Grain boundaries: just vestiges of coarse gamma precipitation zones.
1000.........	1. Intergranular areas: coarse gamma precipitation (sorbitic). Grain boundaries: none visible but broad zones of light etching gamma precipitation still visible.	1. Similar to 900 F, 100 hr but no vestiges of grain boundaries left. Coarse gamma globules in alpha-copper matrix.

Elevated Temperatures:

By testing at the exposure temperature, hardened alloy 25 strip is shown to have quite a different set of values for the properties determined. The tensile strength of hardened strip gradually falls off by exposing it to temperatures up to 500 F. The diminishment of strength in the AT material is at a slower rate than that in the HT condition. In fact, an upward trend is observed at 200 F with the AT material. Because of its prior higher strength, the HT material is stronger than the AT material up to 500 F. However, at 600 to 800 F the AT material loses less strength and is stronger than the HT strip. Above 800 F both have similar strength.

With respect to the elevated-temperature yield strength, the AT and HT materials behave somewhat differently. In the AT condition hardened alloy 25 strip maintains its room-temperature yield strength up to 500 F. A slight inflection is noted at 400 F and a sign of a peak at 500 F. In the HT condition, the yield strength falls off gradually and continuously to 500 F, above which the decrease is much more marked. No inflection or 500 F peak is noted. At 100 to 400 F the yield strength of the HT material is greater than that of the AT material, at 500 F the values are similar, at 600 to 800 F the AT material loses less yield strength than the HT and remains stronger.

TABLE IV.—PER CENT CHANGE IN STRENGTH AS A FUNCTION OF EXPOSURE TIME AT ELEVATED TEMPERATURES.

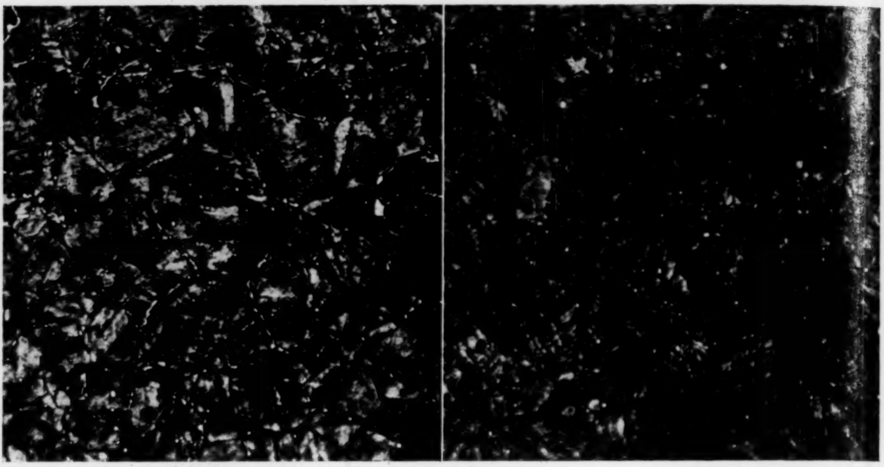
Exposure Temperature, deg Fahr	Exposure Time, hr	Per Cent Change in Strength After Exposure			
		AT Temper		HT Temper	
		Tensile Strength	Yield Strength, 0.2 per cent Offset	Tensile Strength	Yield Strength, 0.2 per cent Offset
Room-Temperature Tests					
100	120	0	−1.8	−1.4	+0.5
200	120	−1.0	0	+3	+2
300	120	0	−1.8	0	+2
400	120	−0.5	−13	+3	+3
500	120	+3	+6	+3	+2
600	120	−12	−10	−10	−15
700	40	−26	−32	−29	−36
800	40	−24	−46	−35	−45
900	20	−27	−47	−25	−44
1000	20	−20	−41	−28	−40
1150	20	−14	−26	−23	−42
Elevated-Temperature Tests					
100	100	+4	+4	+1.0	+1.0
200	100	−1.0	0	−0.5	−0.5
300	100	+7	+2	−0.5	−1.0
400	100	+2	+7	−4	−6
500	100	−2	+3	−1.0	+0.7
600	100	+9	−4	−17	−30
700	100	−30	−26	−32	−42
800	100	−22	−25	−34	−45
900	100	−19	−19	−36	−43
1000	100	−19	−35	−33	−33
1150	100	0	−14	−18	−22

The elevated - temperature proportional limits tend to follow the trend of the elevated - temperature ultimate strengths. A peculiar peak at 200 F is noted along with two drop-offs in the case of the HT material. No explanation of this can be given at this time.

The elevated-temperature elongation values, which are plotted in a reverse manner to the above curves, show a decrease from 100 to 600 F with the AT material and to 400 to 500 F with the HT material. Exposure to temperatures above 600 to 700 F causes a rapid increase in elongation. These effects are the result of further precipitation hardening up to 400 to 600 F and overaging above 600 to 700 F.

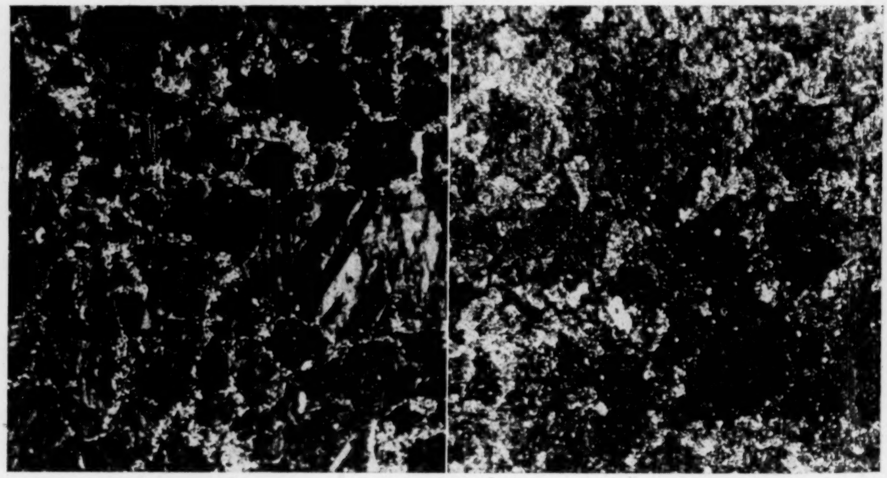
The tensile modulus of elasticity is seen to diminish slowly up to 600 F for the AT material and up to 500 F for the HT. A rapid drop occurs then up to 800 F. Above 800 F the decrease occurs at a slower rate. Exposure time had no particular effect on elevated-temperature modulus values and thus is not considered further.

Again considering Table IV where changes occurring in elevated-temperature strength as a result of prior exposure up to 100 hr at the testing temperature are plotted, we note that the HT material shows stability up to 500 F. However, the AT material demonstrates stability up to 600 F. A prior exposure period of 100 hr generally improves the strength of AT material at any given temperature up to 600 F. On the other hand, a slight diminution is noted with the HT strip. This may indicate that



(*a*) 0 hr at 600 F (×500). Appearance similar to all specimens reheated from room temperature to 500 F.

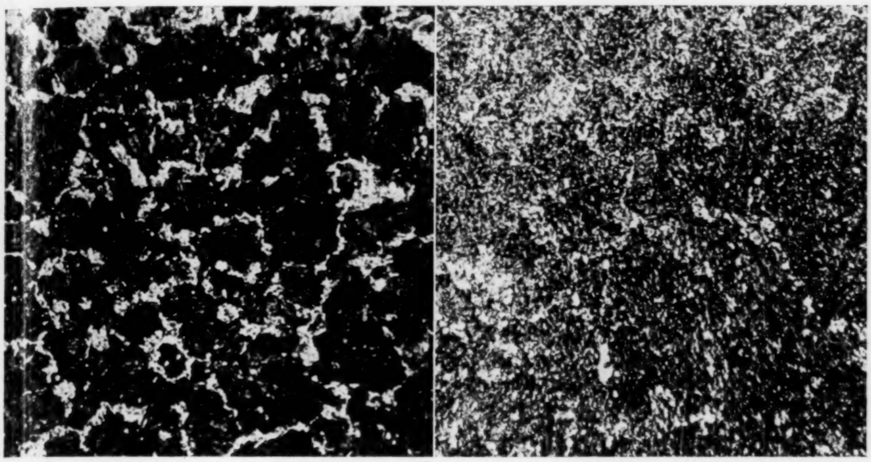
(*b*) 100 hr at 600 F (×500). Not much change from Fig. 10(*a*) but somewhat less distinct structure.



(*c*) 100 hr at 700 F (×500). Note coarse gamma precipitate in broadening intergranular areas.

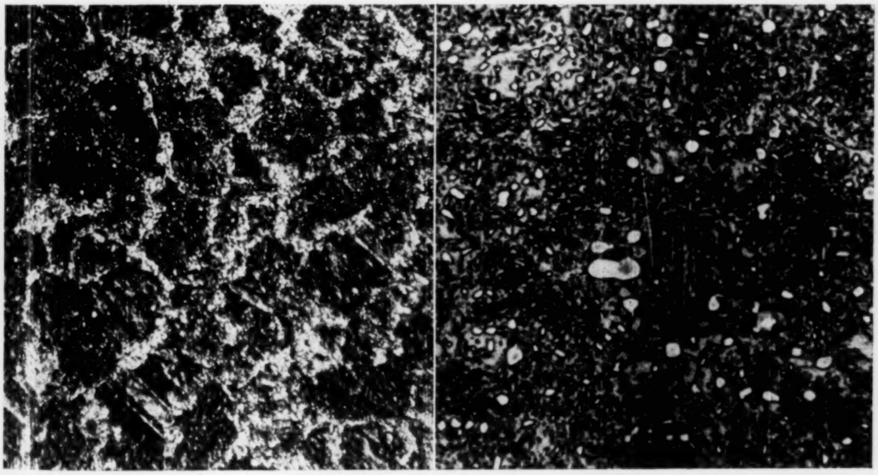
(*d*) 100 hr at 800 F (×500). Note broad intergranular gamma precipitation areas and coarser structure in grains.

FIG. 10.—Microstructure of 25 AT Strip



(*e*) 0 hr at 900 F (×500). Note light etching intergranular areas of coarse gamma precipitate.

(*f*) 100 hr at 900 F (×500). Note coarse "sorbitic" structure with vestiges of grain boundary areas.



(*g*) 0 hr at 1000 F (×500). Note broader intergranular areas of coarse gamma precipitate.

(*h*) 100 hr at 1000 F (×1000). Note general structure of coarse gamma globules in alpha-copper matrix.

Reheated for Various Times and Temperatures.

further precipitation hardening occurs in the somewhat weaker AT material (that is, up to 700 F). A slight stress relieving may occur in the HT strip up to 600 F.

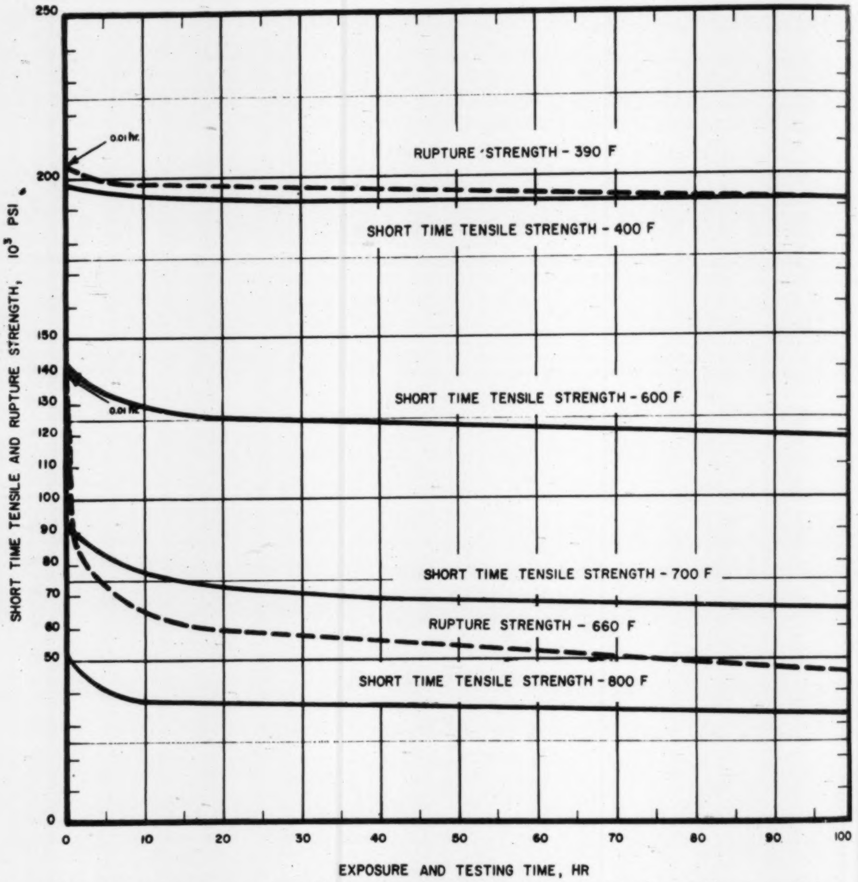


FIG. 11.—Elevated-Temperature Short-Time Tensile Strength of 25 Alloy After Long Exposures to Test Temperature Compared to Corresponding Rupture Strength of Binary Copper-Beryllium (2.25 per cent Beryllium) Alloy. (Data from Parker and Ferguson (3)).

Comparison with Stress-Rupture Properties:

Although no creep and stress-rupture data were found in the literature for alloy 25, Parker and Ferguson (3) reported results of testing copper-beryllium alloy wire containing 2.25 per cent beryllium. This beryllium content is considerably above that of typical alloy 25 which usually ranges between 1.85 and 1.95 per cent beryllium. These stress-rupture data are plotted in Fig. 11 and compared to short-time tensile strength of alloy 25 at corresponding test temperatures and exposure times.

It is seen that the 2.25 per cent beryllium alloy is stronger than alloy 25 on a short-time (0.01 hr) basis both at 390 and 660 F. This would be expected as a result of the greater beryllium content in the wire alloy. This difference is small at 390 F but at 660 F is large. The 660 F stress-rupture strength is 140,000 psi for 0.01 hr, then drops sharply to about 60,000 psi for 20 hr and then decreases slowly with more time. The 660 F stress rupture values are intermediate to short-time tensile strengths measured at 700 and 800 F. Loss in rupture strength as a function of time is minor at 390 F but rapid at 660 F. In both cases, diminution of rupture strength occurs at a faster rate than does short-time tensile strength at comparable exposures. These observations made on rupture and short-time tensile strength would be expected, the greater instability at 660 F being the result of active overaging occurring at this temperature.

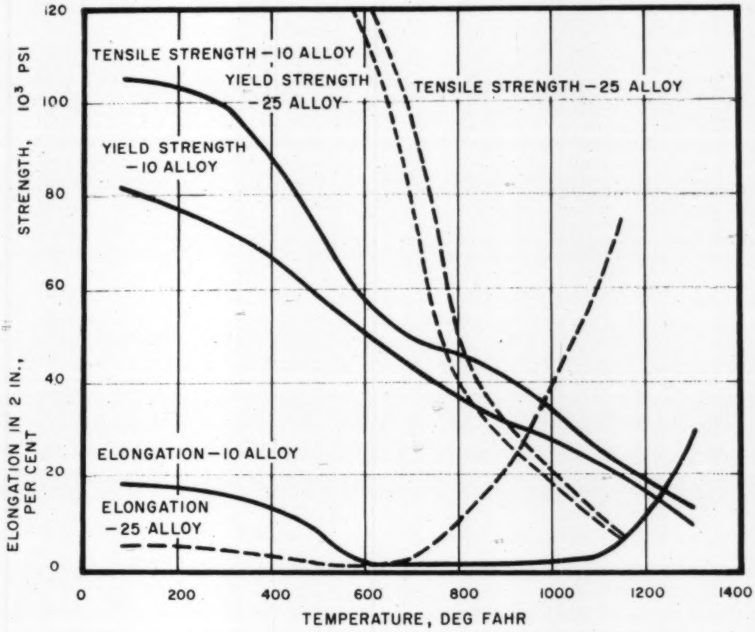


Fig. 12.—Comparison of Elevated-Temperature Tensile Properties of 25 Alloy and 10 Alloy in AT Temper.

Comparison with Elevated-Temperature Tensile Properties of Alloy 10:

It is interesting at this point to compare the elevated-temperature tensile properties of 0.020 in. alloy 25 AT strip with those of 0.020 in. alloy 10 AT strip, which contains 0.45 to 0.65 per cent beryllium, 2.50 per cent cobalt, and balance copper. Alloy 10 hardens by precipitation of cobalt beryllide, and peak hardening occurs by aging from 850 to 950 F; this differs from alloy 25 which hardens by precipitation of copper beryllide (gamma phase) and with maximum hardening occurring at 600 to 650 F. Such a comparison is shown in Fig. 12.

It is seen that alloy 25 AT strip has superior strength to alloy 10 AT strip up to about 825 F. Above this temperature alloy 25 weakens with increasing temperatures more rapidly than alloy 10. Both alloys suffer an elongation minimum at 600 F. Elongation increases above 700 F for alloy 25 AT strip but not until above 1100 F in the case of alloy 10 AT strip.

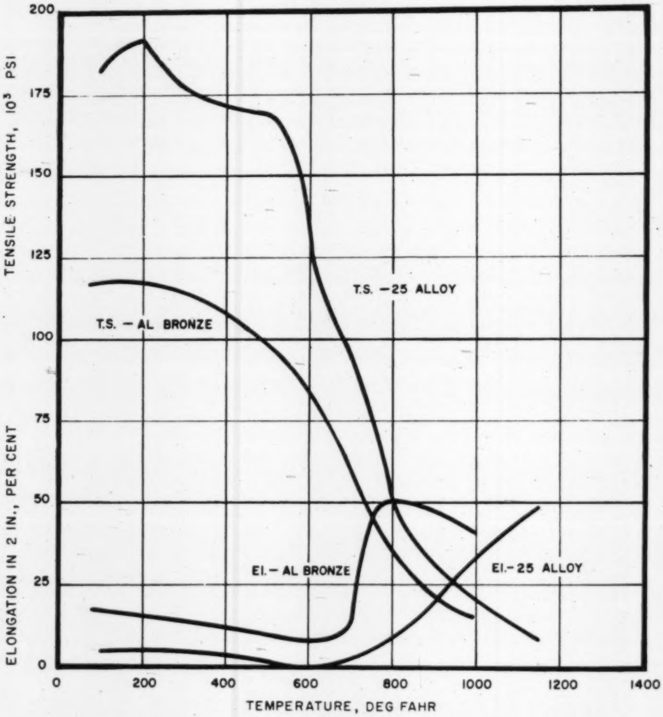


FIG. 13.—Comparison of Elevated-Temperature Tensile Strength and Elongation (per cent in 2 in.) of Alloy 25 AT Strip and Annealed Wrought Aluminum-Bronze Alloy (10Al-3Fe-5Ni).

Comparison with Other Copper-Base Alloys:

The properties determined herein for alloy 25 strip should be compared with those of phosphor bronze strip since both alloys are used in related spring applications (phosphor bronze where design demands are lower and alloy 25 where design demands are higher). However, one cannot usefully compare the elevated-temperature tensile properties of the two alloys. This is because the maximum recommended service temperature of phosphor bronze is only 225 F.

Thus, the wrought copper-base alloy exhibiting the highest elevated-temperature strength in the Upthegrove and Burghoff compilation in 1956 (5) was selected for comparison to alloy 25 AT strip. This alloy contains nominally 10 per cent aluminum, 3 per cent iron, and 5 per cent nickel. The comparison is shown in Fig. 13. Here it is seen that alloy 25 AT strip holds greater than a 65,000 psi superiority in tensile strength over this aluminum bronze up to 500 F. Above this temperature, which is above the service temperature of both alloys, the gap between the strength of both alloys diminishes appreciably. The aluminum bronze exhibits more tensile elongation up to the 600 F minimum; above this temperature this property increases more rapidly than does the elongation of alloy 25.

SUMMARY

In summary it can be said that copper-beryllium alloy 25 demonstrates good stability of tensile properties up through 500 F for the time periods investigated and this could be considered as a limiting service temperature for exposure and use up to 100 to 120 hr. As discussed, it would be necessary to use stress levels significantly below the short-time tensile yield strength or proportional limit in order to avoid excessive creep or the possibility of creep-rupture.

"Secondary hardness" effects were noticed in reheating hardened strip from 100 to 600 F, indicating a renewal of precipitation strengthening effects in both AT and HT material and a possibility of strain hardening during testing of AT material up to 600 F. The original and induced hardening limited elongation up to 600 F. Above 600 F, as a result of overaging and intergranular contributions to flow, hardened alloy 25 strip weakened significantly and exhibited considerable tensile elongation. The AT material was stronger than the HT material above 700 F, indicating somewhat more stability of structure.

This work further demonstrated that maximum hardening can be accomplished either by long-time aging at about 500 F or possibly by a shorter double-aging treatment. Combinations of hardness and ductility to provide improved toughness should be accomplished by overaging at 700 to 800 F. Oxidation in air had no observable deleterious effect on the tensile properties determined.

Creep-rupture, elevated-temperature fatigue, and relaxation studies would better round out the desired elevated-temperature engineering properties of alloy 25 strip for use as springs, contacts, and diaphragms.

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ELEVATED TEMPERATURE CHARACTERISTICS OF TYPE 347 STAINLESS STEEL*

By S. Y. Lu,[1] Marc Trubert,[1] and William A. Nash[1]

Synopsis

The mechanical properties of type 347 stainless steel at rapidly attained elevated temperatures are studied. The investigation is divided into two significant portions. In the first of these the environments considered are: (1) heating to 2200 F in 15 sec, then loading in tension at the strain rate of 1.0 in. per in. per sec, during which phase the effect of various numbers of cycles of heating and cooling prior to loading is also considered; (2) heating to 2200 F at a rate of 200 F per sec with various initial stresses present in the specimen; (3) heating to 2200 F in 15 sec, followed by various lengths of time in which the temperature is held constant prior to loading at a constant strain rate; and (4) thermal cycling between 400 and 2200 F with various initial stresses present in the specimen. In the second part, the effects of various heating rates and peak temperatures on the number of cycles to fracture are studied.

Existing information on mechanical properties of steel at elevated temperatures is concerned with either (*a*) comparatively low strain rates, such as 0.1 in. per in. per sec or less, or (*b*) high strain rates, such as 1000 in. per in. per sec. Comprehensive investigations pertaining to (*a*) for type 301 annealed stainless steel and to (*b*) for mild steel have been carried out by the Southern Research Inst. (**1**)[2] and Nadai and Manjoine (**2**), respectively. A recent book edited by D. W. Grobecker reports on the properties of various metals at elevated temperatures (**3**). Some general information can be found in this book; however, systematic test data are still lacking. The present investigation determined, experimentally, the effects of elevated temperatures and repeated heating on the basic mechanical properties of type 347 annealed stainless steel.

The peak temperatures of the steel specimens ranged from 1800 to 2200 F, while the specimens were heated from room temperature to the peak value in periods of time from 7 sec to 30 sec. During the constant strain rate tests when the coupons were loaded to fracture, the strain rate was approximately 1.0 in. per in. per sec.

Test Apparatus

Test Machine:

The special-purpose test machine was oriented in a vertical plane with four heavy columns—one at each corner.

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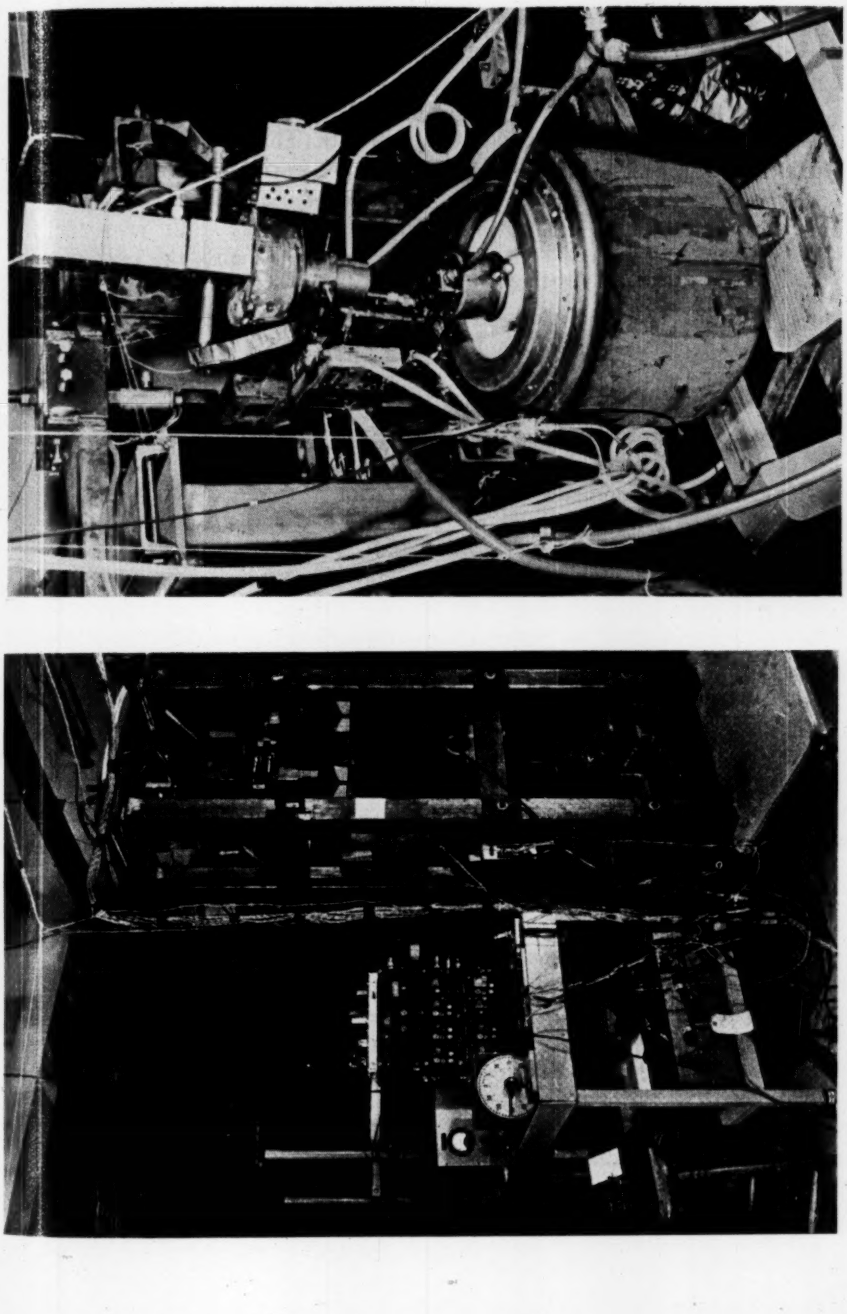
[2] The boldface numbers in parentheses refer to the list of references appended to this paper.



(a) Front view. (b) Back view.

FIG. 1.—Test Machine Used for Determination of Mechanical Properties.

The foundation was a rigid horizontal base provided with casters and leveling screws. A front view of the test machine and some of the instruments are shown in Fig. 1(*a*). The radiofrequency filter boxes, the amplifier in the recording circuit, a timer for thermal cycling, an alarm clock, and the switch box to close the circuit for calibration are on the upper deck of the table. The potentiometer used for thermocouple calibration is on the lower deck. The power switch box is on the stool. Figure 1(*b*) shows the tuning system and the gas-filled capacitor. All the plastic tubes hold running water for cooling the coil. The motor used for the stress-strain test is under the capacitor tank. Figure 2 is a close-up of the central portion of the test machine. The dynamometer, upper grip, specimen, and lower grip can be seen aligned vertically. Three thermocouples are attached by spot welding to the steel specimen and connected to the filter boxes. Lead weights for thermal cycling tests are shown at the bottom part of the picture. The two cantilever-type extensometers can be seen at the right side. A portion of the capacitor tank is shown behind the lower grip.

The axial load in the specimens was measured by a dynamometer equipped with electric strain gages. The upper end of the dynamometer was fixed to the top plate of the test machine. The lower end was connected to the upper grip which, in turn, clamped the upper end of the test specimens. Both the upper and lower grips consisted of pairs of jaws which were tightened by a torque wrench. The teeth of the jaws were unidirectional to prevent slippage between test specimens and grips during tests.

The lower grip was attached to the other end of the test coupon. Provisions were made to apply either dead loads or a constant rate of pulling to the lower grip. For tests involving initial stresses, dead weights were coupled to this grip, while for constant strain rate tests it was lowered by means of a chain drive. The end of this chain passed over a gear which was driven at a uniform angular velocity selected to give the desired constant strain rate. This gear was mechanically linked by a worm-gear drive arrangement to a 5-hp motor with a rated speed of 1070 rpm. Various strain rates could be obtained by changing the gear ratios in the drive system.

To elongate the specimens the motor was started first, then a clutch located between the motor and the worm-gear unit was engaged. This activated the gear, chain, and the lower grip, thereby pulling the test coupons. Both grips were attached to spherical-seat joints to provide as nearly perfect axial alignment as possible. As soon as the specimen was broken, an electric switch was actuated, disengaging the clutch and stopping the motor. Simultaneously, the high-frequency power was turned off.

Heating System:

The induction-heating technique was selected because it made possible the accurate programing of any desired temperature-time relation into the specimens. The induction-heating coils were placed against one surface of the test coupon with a thin layer of mica between the coils and the coupon. The coils functioned as the primary of a transformer, and the specimen as a loaded secondary. The current in the coil induced a voltage in the test coupon, causing a current of large magnitude to flow, thereby inducing heating. The power source was a 20-kw radiofrequency generator with an operating frequency of 2 Mc.

The direct current at high voltage was converted by a rectifier supplied from a

22-kva transformer. The direct current was applied to two type 892 power tubes which supplied energy pulses to a storage circuit. Two gas-filled capacitors were used in this circuit. Their capacitances were 1500 f and 0.06 f. The pressure inside the capacitors was maintained between 130 and 150 psi, and a constant rate of water flow was maintained to cool the tubes and the heating coils.

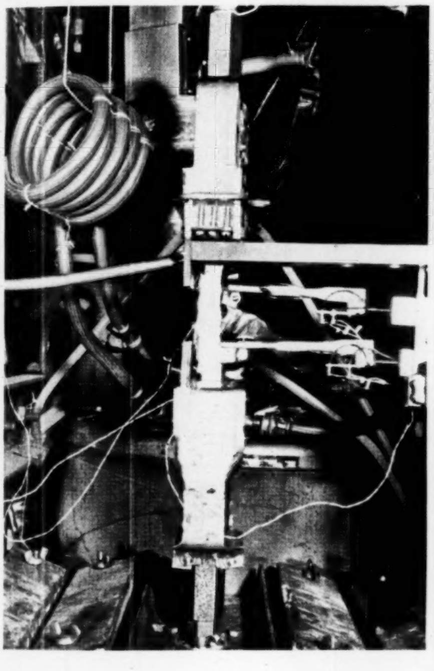


FIG. 2.—Close-up View of Test Machine Showing the Specimen, Dynamometer, Extensometers, and Upper and Lower Grips.

Control System:

Chromel-alumel thermocouples were welded to the steel specimens. The voltage at the cold joint of the thermocouple was amplified, then compared with the voltage produced by the function generator in a d-c amplifier. The function generator was a circuit especially designed to yield a time-dependent voltage. The voltage increased with time to a certain value at the end of the heating period, and remained at that value as long as the system was in operation. The rate of voltage increase was lower in the first half of the heating period than in the second half.

The d-c differential amplifier sensed the voltage difference between the thermocouples and the function generator. When the temperature of the specimens was lower than the peak temperature, the voltage from the function generator was higher than that from the thermocouples; therefore the heating unit kept transferring electrical energy into the specimen. The function generator was turned on or off by a timer which was set to maintain heating or cooling for certain periods of time. A manually operated switch was used to actuate the function generator when a specimen was to be held at peak temperature. Obviously, no power was supplied to the specimen if the function generator was turned off. A block diagram of the control system is shown in Fig. 3.

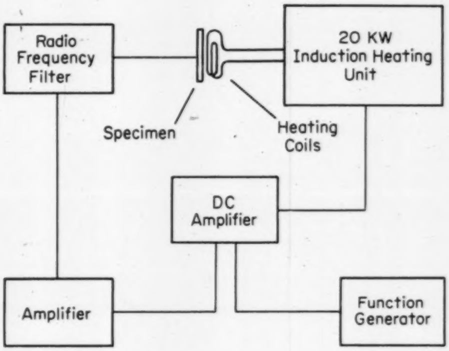


FIG. 3.—Block Diagram of Control System.

Specimens:

Standard size ASTM flat test specimens with a gage length of 2.00 ± 0.02 in. and a nominal width of 0.5 in. were employed. The specimens were made from annealed, type 347 stainless steel sheets. The chemical composition in percentage is listed in Table I. The mean thickness of these stainless steel specimens was 0.063 in. and they had a Rockwell hardness of B 85. Small triangular protrusions, monolithic with the specimens, extended from each end of the gage length so that the elongations could be indicated on a recording oscillograph.

Three chromel-alumel thermocouples were welded on one surface of each specimen. One of the thermocouples served to signal the specimen temperature to the heating system. The other two thermocouples were used in the recording system. A diagram of a specimen appears in Fig. 4.

TABLE I.—CHEMICAL COMPOSITION OF SPECIMENS TESTED.

Element	Per Cent
Carbon, max	0.08
Manganese, max	2.00
Phosphorus, max	0.04
Sulfur, max	0.03
Silicon, max	1.00
Chromium	17.0 to 19.0
Nickel	9.0 to 12.0
Other elements	columbium - tantalum—10X carbon min

Recording System:

The applied load, elongation, and temperature of each test coupon were recorded simultaneously on a multichannel oscillograph. A block diagram of the system is given in Fig. 5.

The electric strain gages in the dynamometer were aligned in the direction of loading. Elongations in the gage length of each test specimen were monitored by means of cantilever-beam type transducers on which the strain gages were mounted. The tip of the beam rested against the small triangular protrusions on the specimen. Although the elongations of the gage length were monitored continuously from the condition of zero stress, the response of strain gages was not recorded on the recording oscillograph until plastic action had begun. All strains indicated anywhere in this study are due solely to the effects of axial

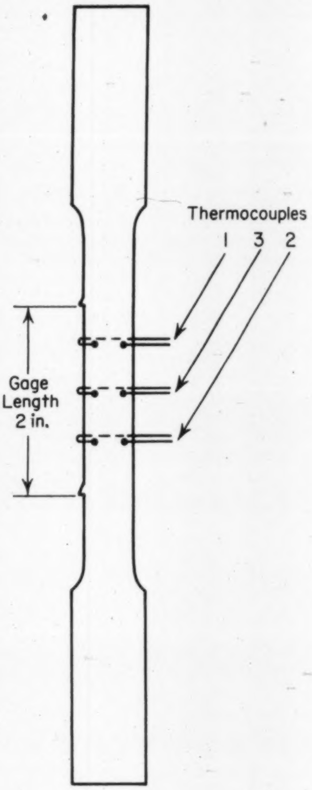


FIG. 4.—Test Specimen Showing Attachment of Thermocouples.

Thermocouples 1 and 2 are for temperature recording; thermocouple 3 is for temperature control.

tension, and do not include the additional elongations arising from thermal expansion. Two thermocouples were used to measure the temperature at two locations on each specimen; thus a mean temperature could be found from the two values recorded. All four signals passed through radiofrequency filters, then through a calibration circuit before the recording circuits were activated. The calibration values were shown as straight lines a certain distance above the reference line on the recording paper. The recorded test data were interpreted with respect to the corresponding calibration line.

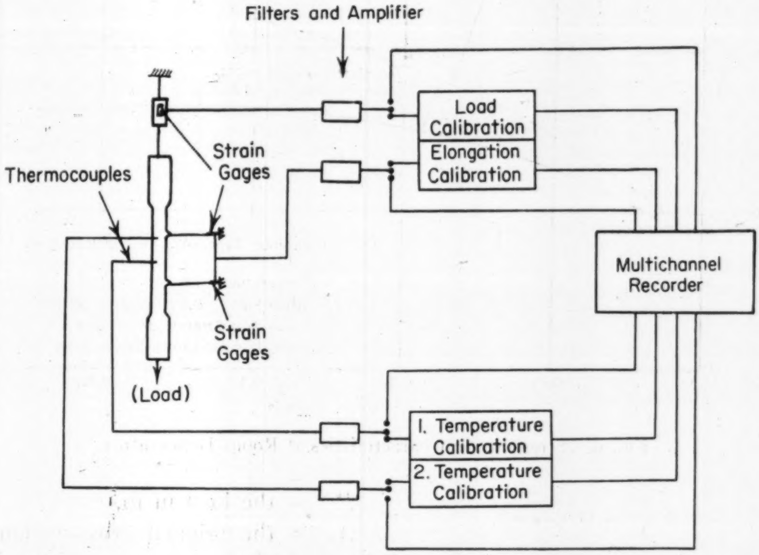

Filters and Amplifier
Load Calibration
Elongation Calibration
Strain Gages
Thermocouples
Multichannel Recorder
Strain Gages
(Load)
1. Temperature Calibration
2. Temperature Calibration

FIG. 5.—Recording System.

TEST PROGRAM AND RESULTS

The program was divided into two significant parts: the first, to determine the basic mechanical properties of type 347 stainless steel at a peak temperature of 2200 F, the second, to study the effects upon its mechanical properties of varying the heating rate and the peak temperature of the steel specimens. The procedures and results of the two parts are discussed below. Generally, three tests were made for each prescribed condition. These results are either bounded within the upper and lower lines in the figures, or averaged to indicate their mean value.

Part I:

This part of the program was further divided into the following four phases:

Phase I.—An investigation of the mechanical properties of the material under various conditions with zero initial stress was conducted during this phase. Room-temperature tension tests were first made to determine the stress-strain characteristics of the material. The results of these tests are indicated on Fig. 6, which shows two stress-strain curves. The upper plot is the true stress versus true strain curve, and the lower one is the nominal stress versus nominal strain curve. These characteristics were determined at a constant strain rate of 0.89 in. per in. per sec.

The equations used to determine the true stress and true strain (4) are:

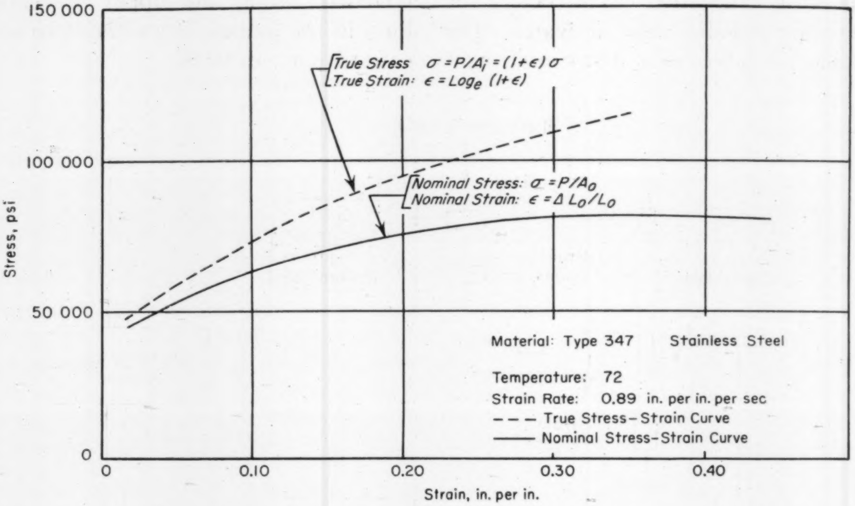


FIG. 6.—Stress-Strain Characteristics at Room Temperature.

$$\bar{\sigma} = \frac{P}{A_i} = (1 + \epsilon)\sigma$$

$$\bar{\epsilon} = \log_e (1 + \epsilon)$$

where:

$\bar{\sigma}$ = the true unit stress in psi,

A_i = the instantaneous cross-sectional area of the test coupon in sq in.,

$\bar{\epsilon}$ = the true strain in in. per in., and

e = 2.718.

These equations are employed only in that region of the stress-strain curve preceding the ultimate or tensile strength of the material. The equations used to determine the nominal stress and nominal strain are:

$$\sigma = \frac{P}{A_0}$$

$$\epsilon = \frac{\Delta L_0}{L_0}$$

where:

σ = the nominal unit stress in psi,

P = the load in lb,

A_0 = the original cross-sectional area of the test coupon in sq in.,

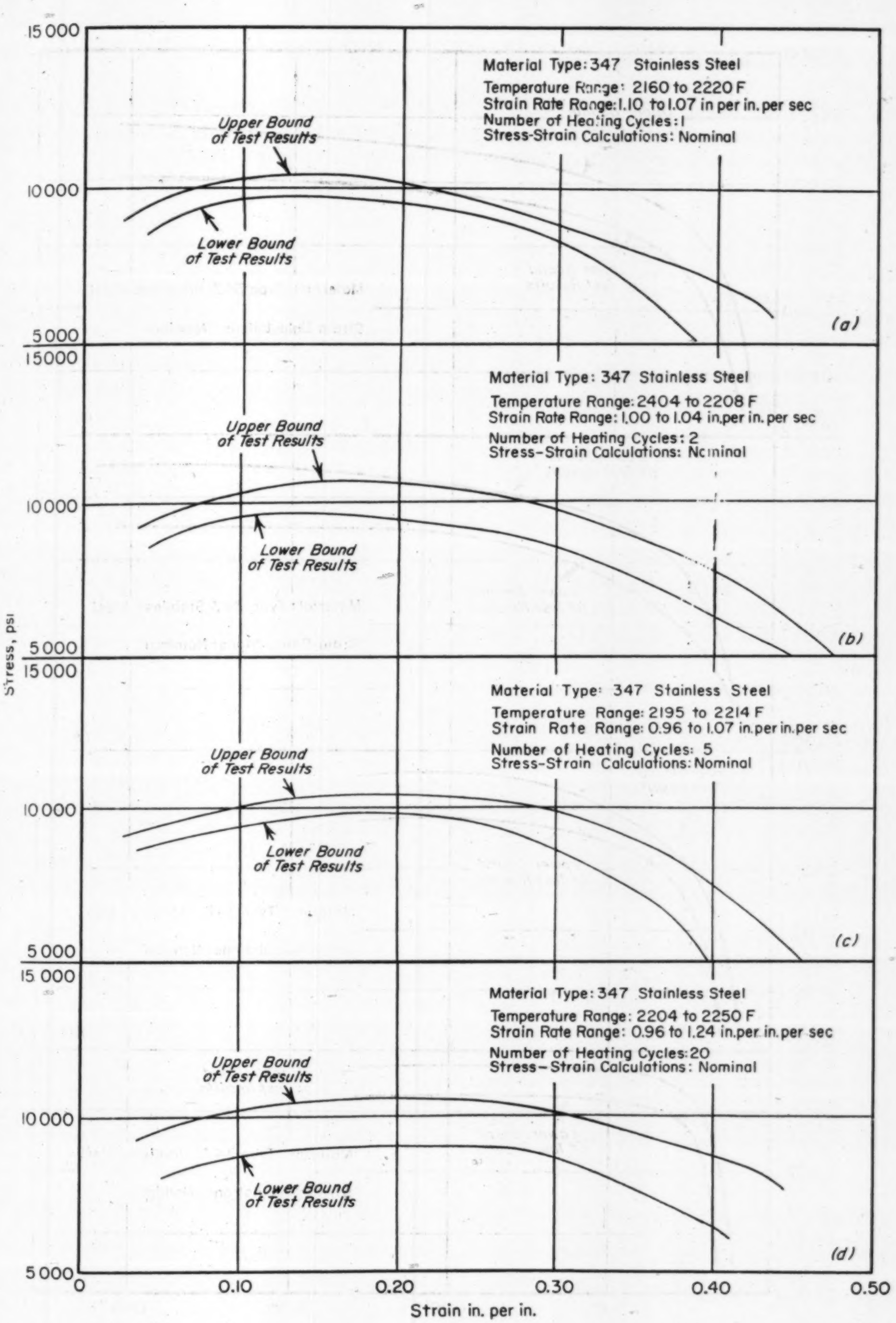
ϵ = the nominal strain in in. per in.,

L_0 = the original gage length of the test specimen in in., and

ΔL_0 = the change in L_0.

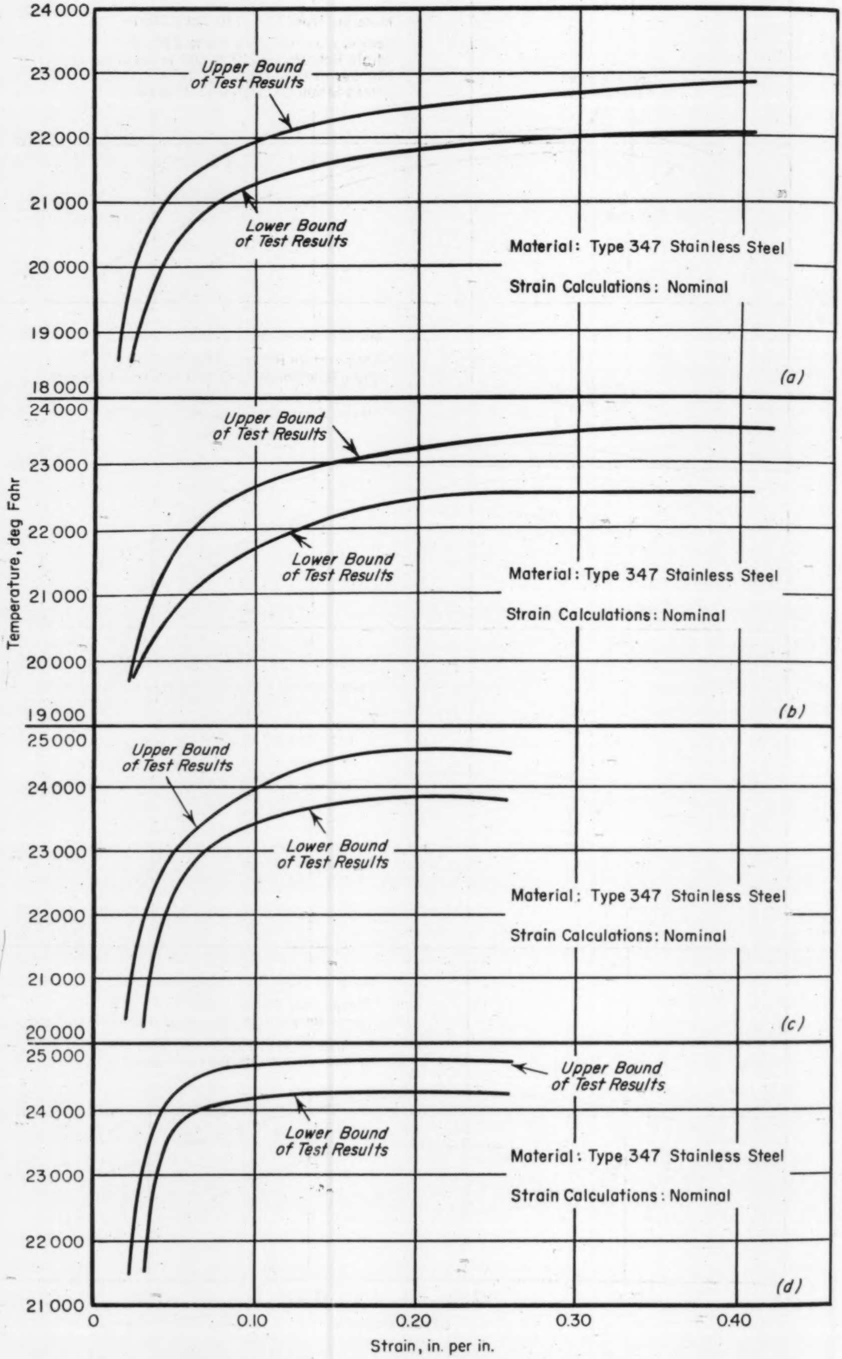
Test coupons were next heated from room temperature to approximately 2200 F in 15 sec. While this temperature was maintained constant, the coupons were loaded to fracture at a constant strain rate of approximately 1.0 in. per in. per sec and the stress-strain characteristics were determined. The results of these tests are shown in Fig. 7(*a*).

The next series of coupons was sub-



(*a*) One heating cycle.
(*b*) Two heating cycles.
(*c*) Five heating cycles.
(*d*) Twenty heating cycles.

FIG. 7.—Stress-Strain Characteristics for Various Heating Cycles; No Initial Stress in Test Coupon.



(*a*) Initial stress, 10,100 psi, equal to the 2200-F tensile strength.
(*b*) Initial stress, 8080 psi, equal to 80 per cent of the 2200-F tensile strength.
(*c*) Initial stress, 6300 psi, equal to 62.4 per cent of the 2200-F tensile strength.
(*d*) Initial stress, 4550 psi, equal to 45.1 per cent of the 2200-F tensile strength.

FIG. 8.—Temperature-Strain Characteristics for Various Initial Stresses in Relation to the 2200-F Tensile Strength.

jected to a temperature rise from room temperature to approximately 2200 F in 15 sec, then cooled to 400 F within 60 sec and immediately reheated to approximately 2200 F in 13 sec. While this temperature was held constant, the test coupons were loaded to fracture at a constant strain rate of approximately 1.0 in. per in. per sec and the stress-strain characteristics were determined. The results of the two-cycle heating are shown in Fig. 7(*b*).

In a similar manner the stress-strain characteristics for 5 and 20 cycles of heating and cooling prior to loading were obtained. The corresponding stress-strain diagrams are shown in Figs. 7(*c*) and (*d*), respectively.

The two curves shown in Figs. 7(*a*) to (*d*) represent extreme bounds on all test results. They are not to be interpreted as representing results of any specific tests. Also indicated in these figures are the temperature ranges and ranges of strain rates for all tests conducted. Nominal stress and strain are the coordinates employed in Fig. 7(*a*) through (*d*).

Phase II.—An investigation of the mechanical properties of the material under conditions of various initial stresses, which were maintained constant during each test, was conducted during this phase. This was accomplished by applying to the specimens (at room temperature) a tensile stress equal to a certain percentage of the tensile strength obtained at a temperature of approximately 2200 F. Then, while this tensile stress was held constant, the temperature of the specimens was raised at a rate of 200 F per sec until fracture occurred.

The average tensile strength at approximately 2200 F, as determined in Phase I of this investigation, was taken as 10,100 psi.

With an initial stress of 10,100 psi applied to the test coupons, the temperature-strain curves shown in Fig. 8(*a*) were obtained. Again, the two curves shown represent upper and lower bounds of all test results.

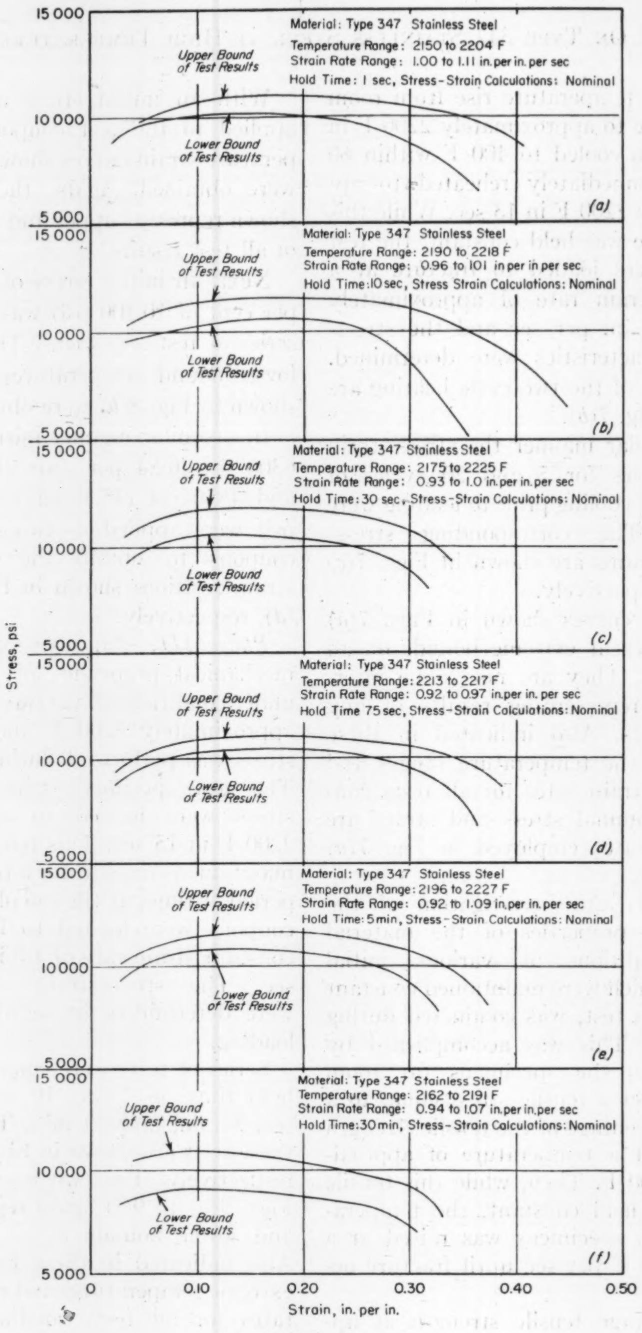
Next, an initial stress of 8080 psi (80 per cent of 10,100 psi) was applied to a series of test specimens. The upper and lower bound temperature-strain curves shown in Fig. 8(*b*) were obtained.

In a similar manner initial stresses of 6300 psi (62.4 per cent of 10,100 psi) and 4550 psi (45.1 per cent of 10,100 psi) were applied to two series of test coupons to obtain the temperature-strain relations shown in Figs. 8(*c*) and (*d*), respectively.

Phase III.—An investigation of the mechanical properties of the material under conditions of various hold times at approximately 2200 F and zero initial stress was performed during this phase. The test specimens with zero initial stress were heated to approximately 2200 F in 15 sec. This temperature was maintained constant for a predetermined period of time; at the end of this time the coupons were loaded to fracture at a constant strain rate of 1.0 in. per in. per sec. The stress-strain characteristics were determined during this period of loading.

Series of tests were conducted for the hold times of 1 sec, 10 sec, 30 sec, 75 sec, 5 min, and 30 min. The results of these tests are shown in Figs. 9(*a*) to (*f*), respectively. The two curves shown in Figs. 9(*a*) to 9(*f*) again represent upper and lower bounds of all test results. Also indicated in these figures are the extreme temperatures and extreme strain rates for all tests conducted for any given hold time.

Phase IV.—The number of thermal cycles required to cause fracture in the



(*a*) **Held for 1 sec.**
(*b*) **Held for 10 sec.**
(*c*) **Held for 30 sec.**
(*d*) **Held for 75 sec.**
(*e*) **Held for 5 min.**
(*f*) **Held for 30 min.**

Fig. 9.—Stress-Strain Characteristics for a Constant Temperature of Approximately 2200 F Held for Varying Times Prior to Loading.

presence of various initial stresses was investigated during this phase. This was accomplished by applying to the specimens (at room temperature) an initial tensile stress equal to a certain percentage of the value of the tensile strength obtained at a temperature of approximately 2200 F. Then the temperature of the specimens was cycled between 2200 and 400 F until fracture occurred. In each cycle the temperature of approximately 2200 F was achieved in 13 sec, and the specimens immediately cooled to 400 F in 60 additional sec. In the first cycle the temperature of 2200 F was attained in 15 sec. The average tensile strength at approximately 2200 F, which was determined in Phase I, was taken as 10,100 psi.

Tests were conducted using initial stresses of 8080 psi (80 per cent of 10,100 psi), 6300 psi (62.4 per cent of 10,100 psi), 4550 psi (45.1 per cent of 10,100 psi), 3800 psi (37.6 per cent of 10,100 psi), and 2800 psi (27.8 per cent of 10,100 psi). Figure 10 indicates the number of thermal cycles required to fracture the test specimens for each of

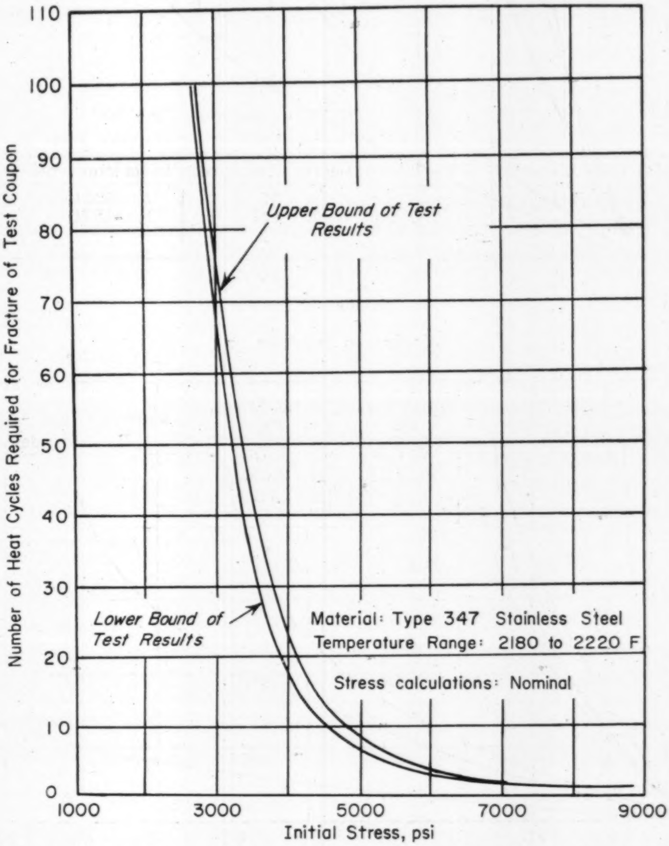
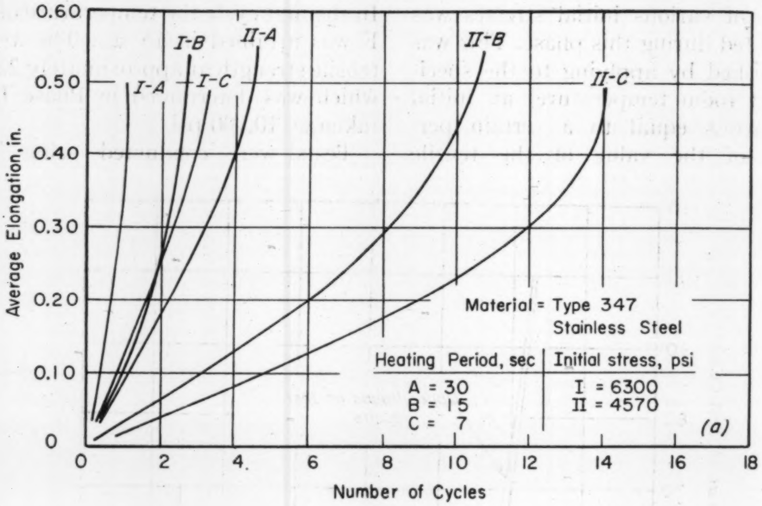
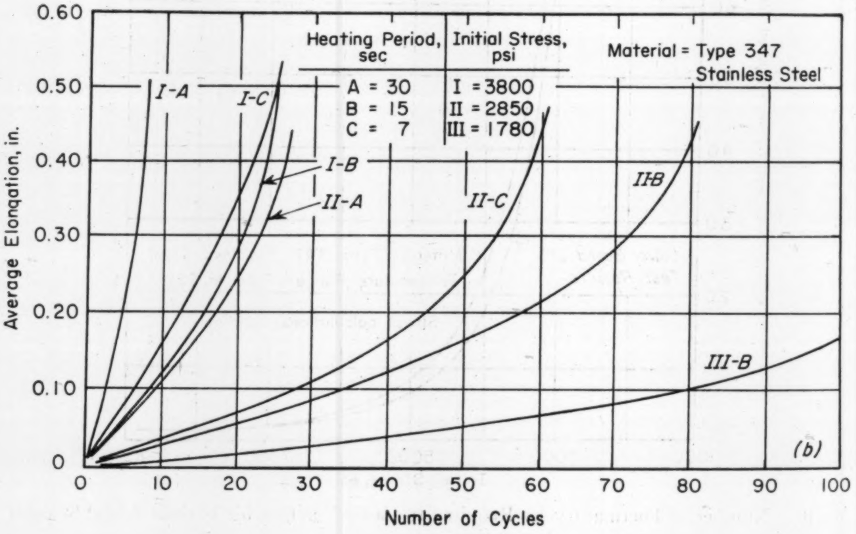


FIG. 10.—Number of Thermal Cycles Required to Cause Fracture for Various Initial Stresses.



(a) Zero to 18 cycles.



(b) Zero to 100 cycles.

FIG. 11.—Elongation in 2 in. Versus Number of Thermal Cycles at Peak Temperature 2200 F.

the various initial stresses. Again, the two curves shown represent upper and lower bounds for all tests.

Part II:

This part of the investigation was directed toward the determination of the effect of (1) the heating rate and (2) peak temperature on the number of thermal cycles required to cause fracture in the presence of various initial stresses. The material was type 347 stainless steel.

Phase I.—Here the peak temperature

was 2200 F. In one series of tests the specimens were heated from room temperature to 2200 F in 7 sec, then cooled to 400 F in 60 additional sec prior to reheating to 2200 F. In another series of tests the specimens were heated from room temperature to 2200 F in 30 sec, after which they were cooled to 400 F in 60 additional sec prior to reheating to 2200 F. In each case thermal cycles corresponding to these temperatures and times were applied until fracture occurred. The elongations between gage points were measured, and these elongations versus the number of cycles, for specimens heated at different rates, are shown in Fig. 11(*a*) and (*b*). The number of heating cycles to failure versus the initial stress is shown in Fig. 12. These graphs were based on the average values of the test data.

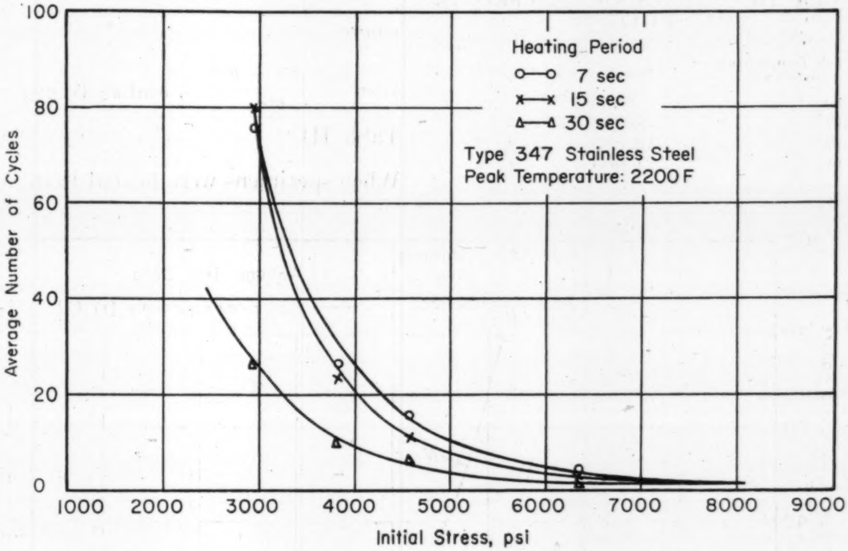


Fig. 12.—Number of Heating Cycles to Failure Versus Initial Stresses at Various Heating Rates.

TABLE II.—AVERAGE NUMBER OF CYCLES TO FAILURE[a] FOR TYPE 347 STAINLESS STEEL.

Nominal Initial Stress, psi	Peak Temperature, deg Fahr		
	1800	2000	2200
1 800....	...	...	>100
2 800....	...	...	80 (90, 80)
3 800....	...	>100	24 (35, 23)
4 400....	...	64 (83, 63)	...
4 600....	...	...	11 (17, 10)
6 300....	>100	18 (21, 17)	3 (3, 3)
8 100....	50 (61, 37)	5 (6, 4)	1 (1, 1)
10 100....	19 (35, 15)	2 (2, 2)	...
11 800....	5 (8, 4)	...	...
13 400....	1 (2, 1)	...	...

[a] Figures inside the parentheses represent the maximum and minimum of the test data, respectively.

Phase II.—Thermal cyclings involving a peak temperature of 2000 or 1800 F were studied. The heating and cooling periods were the same as those used in Phase IV of Part I, that is, 13 sec and 60 sec, respectively, after the first cycle, which involved a time of 15 sec rather than 13. The average test data for thermal cycling at peak temperatures of

1800, 2000, and 2200 F (from Phase IV of Part I) are listed in Table II.

Empirical Relations Based on Phases I and II.—Empirical relations between the number of thermal cycles required to cause fracture and the heating rate, peak temperature, and initial stress were sought. The empirical relations to represent the experimental results have the following form:

Let N = number of thermal cycles required to cause fracture, based upon a heating period of 15 sec. Then

$\theta = \sigma/\sigma_0$

σ = initial stress in the test specimen (applied at room temperature), and

σ_0 = reference stress, depending on peak temperature.

σ_0 is a stress such that when $\sigma = \sigma_0$, N is equal to unity to the nearest integer. It was found that

$$N = \pi_1\pi_2 \qquad (1)$$

where:

$$\pi_1 = \frac{\exp[3(1-\theta^{0.75})]}{\theta^{2.6}}$$

, and π_2 is given in Table III.

TABLE III.—VALUES OF π_2 USED IN CALCULATION.

Peak Temperature, deg Fahr	σ_0, psi	π_2
2200	8 000	1
2000	10 000	$2e^{-\theta^{10}}$
1800	14 000	$4e^{-\theta^5}$

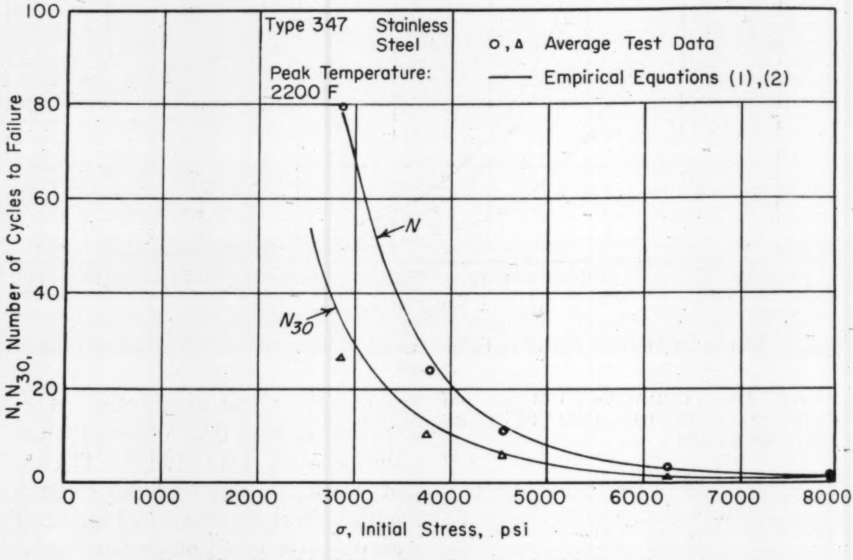


FIG. 13.—Empirical Equations Compared with Test Data.

When specimens were heated from room temperature to 2200 F in less than 15 sec, the number of cycles to fracture was not significantly changed from the number found for the 15-sec period. This can be observed from Fig. 12. From tests using a heating period longer than 15 sec, if N_{30} denotes the number of thermal cycles required to cause fracture at a heating period of 30 sec and peak temperature of 2200 F, then

$$N_{30} = \pi_1 \exp (\theta^{2.5} - 1) \ldots\ldots (2)$$

The above empirical relations as well as all test data are shown in Figs. 13 through 15. These figures indicate that the above relations satisfactorily represent the behavior of the stainless steel in these various environments.

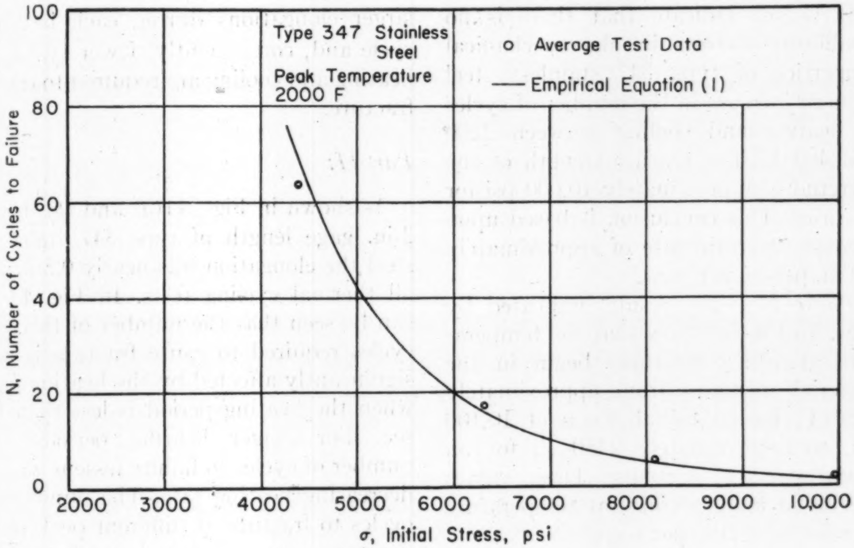


FIG. 14.—Empirical Equation Compared with Test data.

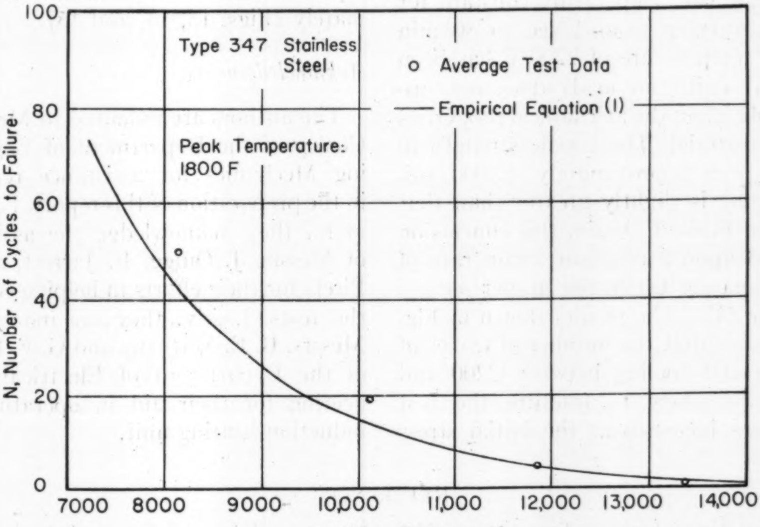


FIG. 15.—Empirical Equation Compared with Test Data.

DISCUSSION OF RESULTS AND CONCLUSIONS

Part I:

Phase I.—The results shown in Figs. 7(*a*) to (*d*) indicate that there is no significant change in the mechanical properties of type 347 stainless steel with an increase in the number of cycles of heating and cooling between 2200 and 400 F. The tensile strength of the material is approximately 10,000 psi for all cases. This conclusion is based upon a constant strain rate of approximately 1.0 in. per in. per sec.

Phase II.—The results indicated by Figs. 8(*a*) to (*d*) show that the temperature at which fractures begin in the material increases from approximately 2150 F, for an initial stress of 10,100 psi, to approximately 2450 F, for an initial stress of 4550 psi. These values are based upon a constant temperature increase of 200 F per sec.

Phase III.—The results shown in Figs. 9(*a*) to (*f*) indicate that the effect of holding the temperature constant for periods varying from 1 sec to 30 min after a temperature of 2200 F has been attained (with no load) does not appreciably affect the mechanical properties of the material. The tensile strength in all cases is approximately 11,000 psi. This value is slightly greater than that found in Phase I. Again, this conclusion is based upon a constant strain rate of approximately 1.0 in. per in. per sec.

Phase IV.—The results shown in Fig. 10 indicate that the number of cycles of heating and cooling between 2200 and 400 F necessary to fracture the test specimens increases as the initial stress is decreased. It is to be observed that the number of cycles increases very rapidly when the initial stress is less than approximately 4500 psi. Stresses greater than approximately 4500 psi cause larger elongations during each heating cycle and, consequently, fewer cycles of heating and cooling are required to cause fracture.

Part II:

As shown in Figs. 11(*a*) and (*b*), for a 2-in. gage length of type 347 stainless steel the elongation was nearly 0.5 in. in all thermal cycling tests. In Fig. 12 it can be seen that the number of thermal cycles required to cause fracture is not significantly affected by the heating rate when the heating period is less than 15 sec. For longer heating periods the number of cycles to failure lessens with a decreasing heating rate. The number of cycles to fracture at different peak temperatures can be found in Table II. Empirical Eqs 1 and 2 are suggested for representing the test results approximately (Figs. 13, 14, and 15).

Acknowledgments:

The authors are indebted to Mr. I. D. Cooley of the Department of Engineering Mechanics for assistance rendered in the preparation of this report. Furthermore, they acknowledge the assistance of Messrs. J. Quigg, B. Jarrett, and A. Pirela for their efforts in helping conduct the tests. Lastly, they are indebted to Messrs. B. E. Mathews and G. F. Perdue of the Department of Electrical Engineering for their aid in operating the induction-heating unit.

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(2) A. Nadai and M. Manjoine, "High-Speed Tension Tests at Elevated Temperatures, Parts II and III," *Journal of Applied Mechanics*, Vol. 63, p. 77, June, 1941.

(3) D. W. Grobecker, Editor, "Metals for Supersonic Aircraft and Missiles," Am. Soc. Metals, Cleveland, Ohio (1958).

(4) A. Nadai, "Theory of Flow and Fracture of Solids," McGraw-Hill Book Co., New York, N. Y. (1950).

SUMMARY OF PROCEEDINGS OF SYMPOSIUM ON EVALUATION OF METALLIC MATERIALS IN DESIGN FOR LOW-TEMPERATURE SERVICE

This Symposium on Evaluation of Metallic Materials in Design for Low-Temperature Service was arranged in response to the increasing interest, particularly on the part of the aerospace industries, in the behavior of materials at cryogenic temperatures.

The Joint Committee on Effect of Temperature on the Properties of Metals sponsored the symposium. The papers were presented on June 27 and 28, 1961, during the ninth, thirteenth, and twenty-second sessions of the Sixty-fourth Annual Meeting of the Society, at Atlantic City, N. J.

Mr. E. M. Kloeblen, Linde Co., served as Symposium Chairman. Mr. F. B. Foley, Metallurgical Consultant, and Mr. G. R. Irwin, U. S. Naval Research Laboratory, served as Session Chairmen and together presided over the three sessions. Mr. Irwin also was moderator of the Panel Discussion that followed the presentation of the papers.

The papers included in the symposium are:

Analysis of Effects of Test Temperature on the Notch Strength of High-Strength Sheet Alloys—Volker Weiss and John G. Sessler
Welded Ferritic Steel Construction for Intermediate Low-Temperature Service—A. A. Wells
Effect of U-Notch Depth on Impact Resistance Under Simple Beam Loading—Donald C. Reymond
Effect of Small Cracks on the Load-Carrying Ability of High-Strength Steel—G. K. Manning
Fracture of High-Strength Sheet Steel Specimens Containing Small Cracks—J. E. Srawley and C. D. Beachem
Tensile and Impact Properties of Cast Stainless Steels at Cryogenic Temperatures—E. R. Hall
Steels of Improved Fracture Toughness—John M. Hodge
Testing Techniques and Evaluation of Materials for Use at Liquid Hydrogen Temperature—R. Markovich and F. Schwartzberg
Correlation of Notched: Unnotched Tensile Ratios with Tensile Fatigue Properties of Complex Welded Joints in High-Strength 300 Series Stainless Steels at Cryogenic Temperatures—J. F. Watson, J. L. Christian, T. T. Tanalski, and A. Hurlich
Factors Influencing Fracture Toughness of Sheet Alloys for Use in Lightweight Cryogenic Tankage—G. B. Espey, M. H. Jones, and W. F. Brown, Jr.
Design of Cryogenic Storage Tanks for Industrial Applications—Herbert W. Marsh
Brittle Fracture Transition of Some Concrete Reinforcing Steels—Allan L. Tarr
Testing and Design Considerations in Brittle Fracture—S. Yukawa
Influence of Sheet Thickness on Sharp-Edge-Notch Properties of a β Titanium Alloy at Room and Low Temperatures—A. J. Repko, M. H. Jones, and W. F. Brown, Jr.
Panel Discussion

These papers with discussion were issued as *ASTM Special Technical Publication No. 302*, entitled "Symposium on Evaluation of Metallic Materials in Design for Low-Temperature Service."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON ELEVATED-TEMPERATURE COMPRESSION TESTING OF SHEET MATERIALS

Compression testing of sheet materials is of great importance in the building of aircraft. Various techniques for compression testing are presented and evaluated in this Symposium on Elevated-Temperature Compression Testing of Sheet Materials, sponsored by the Test Methods Panel and the Panel on Structural Materials for Airframes and Missiles of the ASTM-ASME Joint Committee on Effect of Temperature on the Properties of Metals.

The papers in the symposium were presented on June 29, 1961, during the thirtieth session of the Sixty-fourth Annual Meeting of the Society at Atlantic City, N. J. Mr. H. C. Turner, Convair, presided over the session and, with Mr. J. R. Kattus, Southern Research Inst., served as Symposium Co-chairman.

The papers included in the symposium are:

Introduction—H. C. Turner
An Evaluation of Compression Testing Techniques of Sheet Materials at Elevated Temperatures—George Gerard
Compression Testing of Sheet Materials at Elevated Temperatures—Ralph Papirno and George Gerard
Rapid-Rate Compression Testing of Sheet Materials at High Temperatures—E. C. Bernett and W. W. Gerberich
Evaluation of Test Variables in Determination of Elevated-Temperature Compressive Yield Strength of Magnesium Alloy Sheet—R. W. Fenn, Jr.
Compression and Buckling Characteristics of Annealed and Aged Inconel 718 Nickel-Chromium Alloy at Temperatures up to 1400 F—J. Dubuc and G. Welter
Evaluation of a System for Compression Testing of Sheet Materials at Elevated Temperatures—W. W. Breindel, R. L. Carlson, and F. C. Holden

These papers with discussion were issued as *ASTM Special Technical Publication No. 303*, entitled "Symposium on Elevated-Temperature Compression Testing of Sheet Materials."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON EXTENSION OF SENSITIVITY FOR DETERMINING VARIOUS CONSTITUENTS IN METALS

In order to review the progress in techniques for determination of trace constituents in metals, ASTM Committees E-2 on Emission Spectroscopy and E-3 on Chemical Analysis of Metals sponsored this Symposium on Extension of Sensitivity for Determining Various Constituents in Metals. The papers in the symposium were presented on June 28, 1961, during the twentieth and twenty-fifth sessions of the Sixty-fourth Annual Meeting of the Society at Atlantic City, N. J.

Messrs. L. M. Melnick, U. S. Steel Corp., and N. E. Gordon, Jr., Westinghouse Electric Corp., served as Symposium Co-chairmen and presided over the morning and afternoon sessions respectively.

The papers included in the symposium are:

Separations in Analysis—J. L. Hague
Some Recently Developed Electroanalytical Techniques for Determination of Traces of Metals—Louis Meites
Radioactivation Analysis—A Specific for Trace Element Determinations—G. W. Leddicotte
Ultratrace Emission Spectroscopy—George H. Morrison and R. L. Rupp
Use of the Electron Probe to Measure Low Average but High Local Concentrations—L. S. Birks and R. E. Seebold
Extension of Sensitivity in Analysis of Impurities in Solids by Mass Spectrometry—C. M. Stevens

These papers with discussion were issued as *ASTM Special Technical Publication No. 308*, entitled "Symposium on Extension of Sensitivity for Determining Various Constituents in Metals."

SIGNIFICANCE OF SELECTED ASTM TESTS FOR CEMENT

DISCUSSION OF METHODS OF TEST FOR FALSE SET OF PORTLAND CEMENT*

By W. C. Hansen[1]

The Working Committee on Premature Stiffening was organized by ASTM Committee C-1 on Cement in October, 1953. The assignment was "to define premature stiffening, make a study of the characteristics of this condition, and then prepare a method of testing for premature stiffening."

At the first meeting on February 3, 1954, the committee voted to have its name changed to the Working Committee on False Set and the following was adopted as a definition of false set: "False set is the rapid development of rigidity in a mixed cement paste, without evolution of much heat, which rigidity can be dispelled and plasticity regained by further mixing without addition of water."

At that time, the Federal government and the states of Nebraska and Washington had methods of testing for false set. These were reviewed and others were suggested.

It was recognized at the outset that false set would not be detected if the mixing period in the test method exceeded a few minutes or if the method of testing disturbed the mortar or paste as the test was being made.

The Federal and Nebraska methods required that the operator determine the amount of water to be used in a neat cement by kneading the paste by hand. The results of these tests could accordingly depend very markedly on the experience of the operator.

In the Washington method, the mixing of a mortar was done by hand with a spoon and the water requirement was determined by use of the flow table, which also seemed objectionable.

The committee experimented with what could be considered a modification of the Nebraska and Washington methods. This led to the development and adoption of ASTM Method C 359 – 55 T,[2] in which a mortar is mixed by machine, the penetration of the Vicat needle is measured at four periods, and the mortar is remixed to determine whether or not the workability can be restored.

The members felt that the principal, if not the only, cause of false set was crystallization of gypsum in the paste or mortar which resulted from the dehydration of gypsum during either grinding or storage. The data in Table I demonstrate the ability of dehydrated gypsum to cause stiffening in powdered quartz paste. The data for this table were determined by grinding quartz to about the fineness of cement in a cold mill with gypsum ($CaSO_4 \cdot 2H_2O$), equivalent to 2.0 per cent SO_3; with molding plaster ($CaSO_4 \cdot \frac{1}{2}H_2O$), equivalent to

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* Presented at the Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] Consulting Chemist, Valparaiso, Ind.; Formerly Director, Research Laboratories, Universal Atlas Cement Division, United States Steel Corp., Gary (Buffington Station), Ind.

[2] Tentative Method of Test for False Set of Portland Cement (C 359 – 56 T), 1958 Book of ASTM Standards, Part 4.

2.0 per cent SO_3; and without either gypsum or plaster. These were blended to give mixtures with 0 to 2.0 per cent SO_3 as gypsum and 0 to 2.0 per cent SO_3 as molding plaster. These blends were then tested for false set. The method showed that as little as 0.25 per cent SO_3 as molding plaster produced stiffening in the mortar with quartz flour as the cementing ingredient. It was recognized that such small amounts of dehydrated gypsum in portland cements might not show stiffening because cements combine with appreciable amounts of SO_3 during the mixing period. However, these data indicate that this

TABLE I.—FALSE SET IN QUARTZ PASTES AS DETERMINED BY ASTM METHOD C 359.

SO_3, per cent	Paste Containing Gypsum, Penetration, mm					Paste Containing Molding Plaster, Penetration, mm				
	Initial	5 min	8 min	11 min	Remix	Initial	5 min	8 min	11 min	Remix
0	50+	45	46	44	50+	50+	45	46	44	50+
0.25	47	43	42	42	47	49	40	38	30	50+
0.50	46	44	40	38	46	46	38	27	20	50+
1.00	46	45	41	43	47	48	10	8	7	50+
1.50	47	47	42	40	46	47	5	0	0	50+
2.00	48	44	41	41	48	41	0	0	0	50+

TABLE II.—RESULTS OF EXTRACTION AND FALSE SET TESTS ON TYPE IS CEMENT.

Treatment	Results of Extraction Test					Results of False Set Test with 32 per cent Water				
	Time of Shaking, min	Composition of Extract, moles per liter				Penetration, mm				
		SO_3	$(OH)_2$	CaO	Na_2O[a]	Initial	5 min	8 min	11 min	Remix
CEMENT AS GROUND										
As received	1	0.0245	0.0279	0.0286	0.0238	50+	47	46	46	50+
	3	0.0157	0.0433	0.0279	0.0311					
Aerated 24 hr	1	0.0523	0.0238	0.0589	0.0172	47	44	30	16	50+
	3	0.0401	0.0239	0.0458	0.0182					
Aerated 48 hr	1	0.0533	0.0232	0.0621	0.0144	17	14	5	2	48
	3	0.0431	0.0234	0.0512	0.0153					
CEMENT FROM SILO										
As received	1	0.0233	0.0285	0.0279	0.0239	50+	45	43	37	45
	3	0.0162	0.0338	0.0276	0.0224					
Aerated 24 hr	1	0.0521	0.0190	0.0560	0.0151	50+	47	46	46	50+
	3	0.0433	0.0242	0.0496	0.0179					
Aerated 48 hr	1	0.0538	0.0225	0.0628	0.0135	46	27	12	10	50+
	3	0.0432	0.0229	0.0515	0.0146					

[a] SO_3 at equilibrium for Na_2O of 0.0135 to 0.0311 moles per liter varies from about 0.019 to 0.025 moles per liter.

method should show false set that was severe enough to cause trouble in the field.

One aspect of the problem of false set that has plagued the producer and user, as well as the investigator, is the ability of some cements to become false setting when exposed for some periods of time to the atmosphere. This is shown in Table II. The data for this table were obtained by testing a commercial cement as drawn from the silo, that is, as received, and after exposure in thin layers to the air of the laboratory for 24 and 48 hr. In addition to the test for false set, the cement was extracted with water and the extracts were analyzed for sulfate, hydroxide, calcium oxide and sodium oxide.

The SO_3 contents of the 1- and 3-min. extract from the pastes of the "as received" sample are very much like those required for equilibrium with solutions containing the amounts of alkali found in these extracts. These results and those of the penetration test indicated that the cement as drawn from the silo did not contain much, if any, dehydrated gypsum. However, the results from the extraction tests of the aerated samples showed these cements to be highly supersaturated with respect to gypsum. Since aeration could not dehydrate the gypsum, one has to conclude that the unaerated cement contained dehydrated gypsum. This leads to the conclusion that aeration decreased the rate at which the cement minerals combined with calcium sulfate and this permitted the gypsum to precipitate from the paste and cause false set.

TABLE III.—RANGE OF PENETRATION OF ELEVEN LABORATORIES.

Cement Sample	Range of Penetrations at Period Indicated for Eleven Laboratories, mm				
	Initial	At 5 min	At 8 min	At 11 min	Remixed
A As received	50+	50+	50+	50+	50+
Aerated	50+	50+	36–50+	20–50+	50+
B As received	7–50+	2–5	1–2	0–1	48–50+
Aerated	2–45	1–5	0–1	0–1	48–50+
C As received	45–50+	6–50+	4–50+	3–46	37–50+
Aerated	11–50+	3–47	1–30	1–13	44–50+
D As received	5–50+	3–50+	3–50+	3–50+	35–50+
Aerated	24–50+	0–9	0–6	0–6	46–50+
80% A + 20% B	50+	50+	48–50+	48–50+	50+
60% A + 40% B	50+	50+	48–50+	46–50+	50+
40% A + 60% B	50+	42–50+	12–50+	4–50+	50+
20% A + 80% B	37–50+	2–23	1–5	1–3	50+

AGREEMENT BETWEEN LABORATORIES

Four samples of cement were tested by eleven laboratories in 1954 to determine the degree to which different laboratories could check each other by this method. The cements were as follows:

A. Type I clinker ground with gypsum in a laboratory mill at atmospheric temperature.

B. Commercial type I cement heated until it showed severe false setting properties by the proposed test.

C. Commercial type II cement.

D. Commercial type III cement.

TABLE IV.—RESULTS OF COOPERATIVE TESTS BY ELEVEN LABORATORIES WITH METHOD C 359.[a]

Laboratory No.	Penetrations, mm									
	Sample as Received					Aerated Samples[b]				
	Initial	5 min	8 min	11 min	Remix	Initial	5 min	8 min	11 min	Remix
SAMPLE A—TYPE I—30% WATER										
1, 2, 3, 4, 7	50+	50+	50+	50+	50+	50+	50+	50+	50+	50+
5	50+	50+	50+	50+	50+	50+	50+	50+	47	50+
6	50+	50+	50+	50+	50+	50+	50+	26	20	50+
8	50+	50+	50+	50+	50+	50+	50+	48	30	50+
9	50+	50+	50+	50+	50+	No Tests Made				
10	50+	50+	50+	50+	50+	50+	46	36	36	50+
SAMPLE B—TYPE I—30% WATER										
1	28	3	2	1	50+	7	2	1	0	50+
2	31	3	1	0	50+	10	2	1	0	50+
3	28	5	2	1	50+	11	3	2	1	50+
4	42	4	1	0	50+	6	1	0	0	50+
5	50+	4	1	0	50+	6	3	0	0	50+
6	48	4	1	0	50+	7	3	1	0	50+
7	33	5	2	1	50+	45	5	1	1	50+
8	14	4	1	1	50	6	2	1	1	50+
9	7	3	1	1	50−	No Tests Made				
10	4	2	1	0	48	2	1	0	0	48
11	50	3	1	1	50	37	3	2	1	50+
SAMPLE C—TYPE II—30% WATER										
1	50+	50+	50+	46	50+	50+	42	15	10	50+
2	50+	32	14	6	50+	50+	42	12	8	50+
3	50+	41	18	8	50+	50+	46	12	6	50+
4	50+	47	13	10	50+	50+	40	11	6	50+
5	50+	48	27	12	50+	11	3	1	1	50+
6	50+	47	35	12	50+	50+	45	28	12	50+
7	50+	47	25	12	50+	50+	47	30	13	50+
8	50+	50+	50+	25	50+	50+	20	6	5	50+
9	48	34	10	7	50+	No Tests Made				
10	45	6	4	3	37	47	5	3	3	44
11	50+	4	13	6	50+	50+	43	12	7	50+
SAMPLE D—TYPE III—32% WATER										
1	50+	50+	50+	50+	50+	50+	3	2	2	50+
2	50+	50+	50+	50+	50+	24	9	6	6	50+
3	50+	44	19	8	50+	37	5	3	1	50+
4	48	48	41	33	50+	18	3	3	2	50+
5	50+	50+	48	45	50+	50+	5	4	5	50+
6	50+	26	22	18	50+	50+	1	0	0	50+
7	30	10	10	9	50+	50+	2	1	0	50+
8	50+	50+	48	48	50+	50+	0	0	0	50+
9	8	4	7	6	50	No Tests Made				
10	5	3	3	3	35	50+	1	0	0	46
11	50+	21	10	10	50+	50+	1	2	1	50+

[a] Prior to adoption by ASTM.

[b] Samples A, B and C spread in thin layers in laboratory air for 2 hr; stored in sealed cans for approximately 24 hr. Sample D spread in thin layer in laboratory air for 45 min.

The samples were tested as received and after aeration. In addition, blends of cements A and B were tested. The results of these tests are given in Tables IV and V. The results for the eleven laboratories varied within the following ranges:

Cement A should not have shown false set and none of the laboratories found any indication of false set on the "as received" sample. However, three out of ten found mild stiffening on the "aerated" sample.

Cement B should have shown severe false setting, and only two laboratories obtained an initial penetration of 50+. All laboratories obtained between 0 and 5 mm at the other periods. When this cement was aerated, the initial penetrations, except for two laboratories, were 11 or less, and at the other periods they varied between 0 and 5 mm.

The false setting characteristics of sample C were not known at the outset of the study. Five laboratories obtained penetrations of less than 10 mm at the 11-min period, the penetrations for the other laboratories varied between 10 and 46 mm. Hence, with the exception of the one laboratory that obtained 46 mm, the laboratories obtained from mild to severe false setting characteristics. On the aerated sample, all penetrations at the 11-min period varied between 13 and 1 mm. It appears, therefore, that the variations of 46 to 3 mm obtained on the "as received" samples were obtained because of different degrees of aeration of the sample. For example, the laboratory that obtained a penetration of 46 mm on the "as received" sample obtained 10 mm on the aerated sample. Laboratory No. 10 obtained initial penetrations of 47 and 45 mm and 11-min penetrations of 3 mm on the "as received" and "aerated" samples, respectively. Hence, it appears that their "as received" sample was badly aerated.

TABLE V.—RESULTS OF COOPERATIVE TESTS BY TEN LABORATORIES WITH METHOD C 359[a] ON BLENDS OF CEMENTS A AND B.

Laboratory No.[b]	Penetrations, mm				
	Initial	5 min	8 min	11 min	Remix
80A + 20B					
All except 10	50+	50+	50+	50+	50+
10	50+	50+	48	48	50+
60A + 40B					
All except 10	50+	50+	50+	50+	50+
10	50+	50+	48	46	50+
40A + 60B					
1, 2, 5, 8, 11	50+	50+	50+	50+	50+
3	50+	50+	46	44	50+
4	50+	50+	50+	49	50+
7	50+	50+	49	48	50+
9	50+	50+	47	48	50+
10	50+	42	12	8	50+
1—Aerated sample	50+	50+	17	4	50+
20A + 80B					
1	50+	11	4	3	50+
2	50+	7	4	3	50+
3	50+	6	4	3	50+
4	50+	12	5	3	50+
5	50+	8	4	3	50+
7	50+	5	3	2	50+
8	50+	5	3	2	50+
9	50+	10	3	2	50+
10	50+	2	1	1	50+
11	50+	23	4	3	50+

[a] Prior to adoption by ASTM.

[b] Laboratory No. 6 did not take part in all tests.

What has been said for sample C applies to sample D. The 11-min penetrations on the aerated sample varied between 6 and 0 mm, whereas on the "as received" sample they varied between 50+ and 3 mm. This certainly appears to be a case of the different laboratories testing "as received" samples that had been aerated to different degrees.

The results with cements A and B agreed fairly well between laboratories.

The first was a non- and the second was a false-setting cement. These were blended to give the four cements for which data are given in Table V. With the 80A + 20B and with the 60A + 40B blend, only laboratory No. 10 obtained less than 50 mm penetration at any period. That laboratory obtained 48 and 46 mm, respectively, at the 11-min period.

With the 40A + 60B blend, except for laboratory No. 10, the penetrations at the 11-min period varied between 50+ and 44 mm. Laboratory No. 10 obtained 8 mm at that period. Laboratory No. 1 aerated the sample and obtained 4 mm at the 11-min period. It appears from this that laboratory No. 10 tended to aerate the samples much more than did the other laboratories.

With the 20A + 80B blend, the penetrations at the 11-min period for all laboratories varied between 1 and 3 mm.

The conclusions from this study were that the results from different laboratories would agree very well if the samples were aerated to the same degree before being tested. Investigators using the Federal Method which required mixing by hand, which is now ASTM Method C 451 - 60 T[3] found that aeration also changed the false setting characteristics as determined by that method. Hence, the two methods appeared to be equivalent in indicating false set and variations caused by aeration.

One advantage of Method C 451 - 60 T, compared to Method C 359 - 56 T, is that the former requires almost no equipment whereas the latter requires a mechanical mixer. On the other hand, with Method C 451, it is often necessary to make several trial batches to obtain the water requirement, whereas with Method C 359 only one batch of 600 g of cement is required for a test. Another advantage of Method C 359 over Method C 451 is that a relatively inexperienced operator can perform it, whereas with the latter an operator must have a large amount of experience to be able to obtain consistent results for the water requirement of a badly false-setting cement.

One sample of cement that showed false set on a paving job was tested by eleven members of the committee. The initial penetrations varied between 50+ and 12; between 14 and 0 at 5 min; between 2 and 0 at 8 min; and between 0 and 1 mm at 11 min. All except two laboratories obtained 50+ on the remix. The two obtained 17 and 20 mm.

During the period of 1956 to 1959, four members of the committee have submitted data on samples from field projects in which trouble was encountered with false set. In some cases, it is not certain that the sample sent to the laboratory was from cement that was actually producing false set in the field. A total of 22 samples was tested. Of these, 2 had penetrations at 11 min of 45 and 47 mm. Four had penetrations at 11 min between 8 and 12 mm and the remaining 16 had penetrations at 11 min ranging between 0 and 3 mm.

It appears from these results that cements that cause difficulties in the field from the standpoint of false set are likely to have penetrations of less than 5 mm at the 11-min period.

SUMMARY

The data in the files of the Working Committee on False Set show good agreement between the results obtained in different laboratories with ASTM Method C 359 on samples from the same lots of cements. These data also show[4] that some cements develop false set when aerated and that erratic results may be obtained unless the operator is careful in the handling of his samples prior to testing. The working committee is currently carrying out an investigation to determine agreement between results obtained with Methods C 359 and C 451.

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[3] Method of Test for False Set of Portland Cement (C 451-60 T), 1960 Supplement to Book of ASTM Standards, Part 4.

SIGNIFICANCE OF TEST FOR CALCIUM SULFATE IN HYDRATED PORTLAND-CEMENT MORTAR*

BY THOMAS B. KENNEDY[1]

"Portland cement is the product obtained by pulverizing clinker consisting essentially of hydraulic calcium silicates, to which no additions have been made subsequent to calcination other than water and/or untreated calcium sulfate, etc." The definition, quoted in part, is taken from ASTM Specifications for Portland Cement (C 150).

The use of calcium sulfate, usually gypsum, in portland cement to control setting goes back to the advent of the rotary kiln. The tendency of early cements toward flash set created a problem. W. C. Hansen (**1**),[2] drawing from Lesley (**2**), attributes the first use of calcium sulfate to control set to workmen in France who added plaster to the mixing water to retard setting when making concrete sidewalks.

It was recognized that while calcium sulfate (gypsum) was valuable in controlling set, too much sulfate could cause excessive expansion and disintegration. Therefore, specification limits on SO_3 were set at 2.0 per cent maximum in 1917 for all cements and remained at this level for many years. In 1930, a tentative specification for high-early strength cement was issued permitting a maximum of 2.5 per cent SO_3.

William Lerch (**3**), at the Annual Meeting of the Society in June, 1946, presented his monumental work on the influence of gypsum on the behavior and properties of portland-cement pastes. Lerch's study was undertaken in the belief that the quantity of gypsum required to regulate properly the setting and hydration of cement would vary with fineness and composition. His findings fully confirmed these suppositions. Lerch found that gypsum acts to retard cements that are flash setting and to accelerate those that are abnormally slow setting. He found that gypsum used in proper amounts minimized shrinkage from drying and expansion from storage in water. He also found higher strengths resulting from use of adequate gypsum. His work went far toward relieving fear of the consequences of using too much gypsum. The beneficial action of gypsum seems to be related to the control of the rate of solution and hydration of tricalcium aluminate (C_3A). Calcium hydroxide also exerts a restraining effect on the reaction, but gypsum and lime together exert more effect than either separately. More gypsum is required in the presence of sodium and potassium alkalies since they accelerate solution of C_3A.

The product of reaction between C_3A

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* Presented at the Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] Chief, Concrete Division, U. S. Army Engineer Waterways Experiment Station, Jackson, Mississippi. Also Chairman, Working Committee on Sulfate Content, ASTM Committee C-1 on Cement.

[2] The boldface numbers in parentheses refer to the list of references appended on this paper.

and gypsum is a calcium sulfoaluminate which occupies greater volume than the sum of its separate components; however, if the reaction goes to completion within an early age—for example, 24 hr at normal temperatures—the paste is sufficiently plastic to accommodate the volume change without damage. In fact, measurable volume change is minimized. If an excess of unused SO_4 ions remain after about 24 hr, subsequent expansion might occur if water enters the concrete. This results from the formation of more sulfoaluminate. Lerch concluded that when gypsum is present in a correct amount, a water extract of hardened cement-sand mortar at 18 hr should contain at least 1.0 g SO_3 per liter, but less than 0.07 g per liter at 24 hr. His work appeared to offer a feasible analytical chemical means for determining when the optimum amount of gypsum had been added to the cement.

DEVELOPMENT OF ASTM METHOD C 265

As a result of the work by Lerch, ASTM Committee C-1 on Cement in 1946 established the Working Committee on SO_3 Content to consider the effects of SO_3 in cement. The working committee made its first report in 1950 (4). The report covered findings of a cooperative investigation wherein the members tested different clinkers ground with different amounts of gypsum. Extraction tests indicated agreement as to optimum with measurements for strength and volume change.

Reproducibility between laboratories was not excellent, but good enough for the working committee to recommend to the Society the adoption of maximum permissible limits of SO_3 based on an extraction test at 24 hr. ASTM Method C 265 – 51 T, Tentative Method of Test for Calcium Sulfate in Hydrated Portland Cement Mortar, was adopted and C 150 – 52 T was issued with provision for limiting SO_3 based on the extraction test. A weakness of this approach was that, although safeguards were provided to prevent too much sulfate, no means was provided to insure use of enough gypsum.

Experience of the cement industry operating under the specification for maximum SO_4 based on extraction at 24 hr was generally unsatisfactory. A high degree of skill was required in performance of the test. In effect, the manufacturer was trying to add enough gypsum so that there would be a little SO_3 left at 18 hr, but practically none left at 24 hr. The analysis was tricky and depended on very close timing and temperature control and strict adherence to detail.

The ASTM example was followed outside the United States, and in 1954 Mexican Standard Specification DGN C-1 adopted limits based on the extraction test, where water extracts of mortars must contain a minimum of 0.1 g SO_3 per liter of extract at 18 hr and not over 0.5 g per liter at 24 hr.

In 1956, the Society again revised the cement specification, adopting limits for SO_3 based on type and composition. Limits on SO_3 have been raised from time to time so that now as much as 4 per cent SO_3 is permitted in type III cement when the C_3A is 8 per cent or more.

Because of difficulties with ASTM Method C 265, another series of cooperative tests was undertaken in the fall and winter of 1955–1956. Five cements with wide range in composition were tested. Test requirements for time between mixing and extraction and on temperature control were made quite restrictive. Analyses for SO_3 were made on extracts taken at both $24 - \frac{1}{4}$ and $24 + \frac{1}{4}$ hr. Reproducibility between laboratories continued to be poor. The high-alkali cements gave the most trouble.

To date, ASTM Method C 265 has been revised five times since originally issued. It is presently designated C 265 – 58 T and contains a sentence in its scope: "This method of test is intended for use primarily by manufacturers of portland cement and those interested in research on a suitable method for determining whether calcium sulfate has or has not been used in an amount considered to be optimum." As its scope indicates, it is considered more suitable for research than for specification purposes.

Before preparing this report, a questionnaire was circulated to each cement manufacturer of ASTM Committee C-1 asking the four following questions:

1. Do you now use ASTM Method C 265 to control SO_3 as near optimum as present cement specifications will permit?
2. Have you ever used ASTM Method C 265 for control of optimum amount of SO_3 within specification limits?
3. Do you consider ASTM Method C 265 practical for control of optimum SO_3 content?
4. Have you any suggestions that would make ASTM Method C 265 a more practical method of test?

Seventy-nine per cent returned the questionnaire, indicating much interest in the problem of sulfate control. None returning the questionnaire said they now use ASTM Method C 265 for control in manufacture. Twenty-four per cent said they had tried the method for control, but only 10 per cent said they considered it practical for control of sulfate. No specific suggestions were received for making the method a more practical workable tool.

Those responding to the questionnaire were generous in their comments about ASTM Method C 265. There seemed to be a general feeling that the poor reproducibility of the method and the length of time required to perform the test prevents its use as a practical tool to control SO_3; however, a number felt the method had considerable value in enabling the manufacturer to discover the optimum sulfate level for his production and work toward it within specification limits.

The Working Committee on SO_3 Content of ASTM Committee C-1 is endeavoring by tests other than Method C 265 to find means for determining optimum SO_3, and at the suggestion of Bailey Tremper of the California Division of Highways, it investigated a method based on early expansion, limited drying shrinkage, and 24-hr compressive strength. These results are reported in the appendix to the Annual Report of Committee C-1 for 1959. At the 1959 Annual Meeting of ASTM, Tremper (5) reported on Cooperative Studies of the expansion and contraction tests. These were conducted by the California Division of Highways and 13 cement mills in California.

The working committee is currently investigating the feasibility of using the 24-hr strength of 2-in. mortar cubes for indicating optimum sulfate. A test of this type would be very attractive because of its simplicity and short time required for results.

SUMMARY

In summary, the work of William Lerch in 1946 on the effects of SO_3 on the properties of hardened cement pastes and mortars indicated best strength and minimum volume change when gypsum was added to cement in optimum amount. The optimum amount appeared to be that which would provide SO_4 ions for combination with C_3A up to about 24 hr at room temperature. Chemical analysis of water extracts of sand-cement mortar for dissolved sulfate at approximately 24 hr appeared to be a practical means of determining when optimum SO_3 had been used.

In 1946, ASTM Committee C-1 established a Working Committee on SO_3 Content. In 1951, ASTM Method C 265 - 51 T was issued. Experience with the method has shown it to have poor reproducibility between laboratories. Many cement manufacturers use this method to establish the best sulfate level for their cements, then manufacture as near optimum as permitted by the specifications. ASTM Method C 265 is rarely used for day-to-day control.

Other methods based on volume change or 24-hr strength of mortar may provide more practical means for determining optimum sulfate and these are being investigated.

REFERENCES

(1) W. C. Hansen, "The Properties of Gypsum and the Role of Calcium Sulfate in Portland Cement," ASTM BULLETIN, No. 212, Feb., 1956, p. 66.

(2) R. W. Lesley, "History of the Portland Cement Industry of the United States," International Trade Press, Inc., Chicago, Ill. (1924).

(3) William Lerch, "The Influence of Gypsum on the Hydration and Properties of Portland Cement Pastes," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 1252 (1946).

(4) H. S. Meissner, "The Optimum Gypsum Content of Portland Cement," ASTM BULLETIN, No. 169, Oct., 1950, p. 39 (TP 225).

(5) Bailey Tremper, "Control of Gypsum in Portland Cement," *Proceedings*, Am. Soc. Testing Mats., Vol. 59, p. 1101 (1959).

DISCUSSION

MR. W. C. HANSEN[1] (*presented in written form*).—Mr. Kennedy states "The product of the reaction between tricalcium aluminate and gypsum is a calcium sulfoaluminate which occupies greater volume than the sum of its components; however, if the reaction goes to completion within early ages—for example, 24 hr at normal temperatures—the paste is sufficiently plastic to accommodate the volume change without damage." I believe that Mr. Kennedy could cite a number of references that would support this statement. However, I also believe that this statement is incorrect and I shall attempt to present data to support this belief.

In 1952, I pointed out that the theory which postulated that the cement minerals dissolve congruently in water to form supersaturated solutions from which the reaction products crystallized appeared to be incorrect (**1**).[2] This theory has been referred to as the "through solution theory." I suggested that the cement minerals could react as solids with water and with materials dissolved in the water to be transformed directly into other solids. In 1955, Jeffrey (**2**) referred to this type of reaction as a solid state reaction and discussed a number of such reactions. Two years ago, I reviewed some of the work of other investigators who appeared to find the through solution theory inadequate (**3**). Very recently, Powers (4) published data obtained by Steinour which showed that cement pastes dilate at ages of about 1½ hr and interpreted this as evidence that the gel formed on the surfaces of the crystals was being pushed away from those surfaces. I propose here to examine the above statement by Mr. Kennedy in terms of these two theories.

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[1] Consulting Chemist, Valparaiso, Ind.

[2] The boldface numbers in parentheses refer to the list of references appended to this discussion.

Table I gives data for the molecular weights, densities, and volume per molecular weight for compounds which will be referred to later. These data were taken from sources that are believed to contain the most reliable data for the densities. Table II gives in Eqs 1, 2, 3, and 4, four reactions that are generally

TABLE I.—MOLECULAR WEIGHTS AND DENSITIES OF COMPOUNDS REFERRED TO IN TABLE II.

Formula of Compound	Molecular Weight	Density	Volume per molecular weight, cu cm
$CaSO_4 \cdot 2H_2O$	172.1	2.32	74.1
$3CaO \cdot Al_2O_3$	270.2	3.04	88.8
$3CaO \cdot SiO_2$	228.0	3.15	72.3
$2CaO \cdot SiO_2$	172.0	3.27	52.6
$Ca(OH)_2$	74.0	2.24	33.2
H_2O	18.0	1.00	18.0
$4CaO \cdot Al_2O_3 \cdot 13H_2O$	560.2	2.08	269.2
$3CaO \cdot 2SiO_2 \cdot 3H_2O$	342.0	2.73	125.3
$3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 31H_2O$	1236.5	1.73	714.7
$3CaO \cdot Al_2O_3 \cdot CaSO_4 \cdot 12H_2O$	622.3	1.95	319.1
$3CaO \cdot Al_2O_3 \cdot 6H_2O$	378.2	2.52	150.0

TABLE II.—DATA FOR POSSIBLE REACTIONS IN PORTLAND-CEMENT PASTES.

	TRICALCIUM ALUMINATE
Eq 1	$3CaO \cdot Al_2O_3 + 3CaSO_4 \cdot 2H_2O + 25H_2O = 3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 31H_2O$
Volume, cu cm	88.8 222.3 450.0 714.7 + (46.4)
Ratio, original volume to expanded	1 8.0
Eq 2	$3CaO \cdot Al_2O_3 + CaSO_4 \cdot 2H_2O + 10H_2O = 3CaO \cdot Al_2O_3 \cdot CaSO_4 \cdot 12H_2O$
Volume, cu cm	88.8 74.1 180.0 319.1 + (23.8)
Ratio, original volume to expanded	1 3.6
Eq 3	$3CaO \cdot Al_2O_3 + Ca(OH)_2 + 12H_2O = 4CaO \cdot Al_2O_3 \cdot 13H_2O$
Volume, cu cm	88.8 33.2 216.0 269.2 + (68.8)
Ratio, original volume to expanded	1 3.0
Eq 4	$3CaO \cdot Al_2O_3 + 6H_2O = 3CaO \cdot Al_2O_3 \cdot 6H_2O$
Volume, cu cm	88.8 108.0 150.0 + (46.8)
Ratio, original volume to expanded	1 1.69
	TRICALCIUM SILICATE
Eq 5	$2(3CaO \cdot SiO_2) + 6H_2O = 3CaO \cdot 2SiO_2 \cdot 3H_2O + 3Ca(OH)_2$
Volume, cu cm	144.6 108.0 125.3 99.6 + (27.7)
Ratio, original volume to expanded	1 1.55
	DICALCIUM SILICATE
Eq 6	$2(2CaO \cdot SiO_2) + 4H_2O = 3CaO \cdot 2SiO_2 \cdot 3H_2O + Ca(OH)_2$
Volume, cu cm	105.2 72.0 125.3 33.2 + (18.7)
Ratio, original volume to expanded	1 1.51

accepted as reactions that may occur with tricalcium aluminate in cement pastes. It also gives in Eqs 5 and 6, the commonly accepted reactions for tri- and dicalcium silicates in cement pastes. The numbers immediately under the formula for the compound in these equations is the volume in cubic centimeters of the grams of the compound taking part, or the reaction products shown in these equations, the volume of the paste should contract and not expand. It is, of course, well known that portland-cement products expand for relatively long periods of time if stored under highly humid conditions. This expansion is not compatible with the through solution theory.

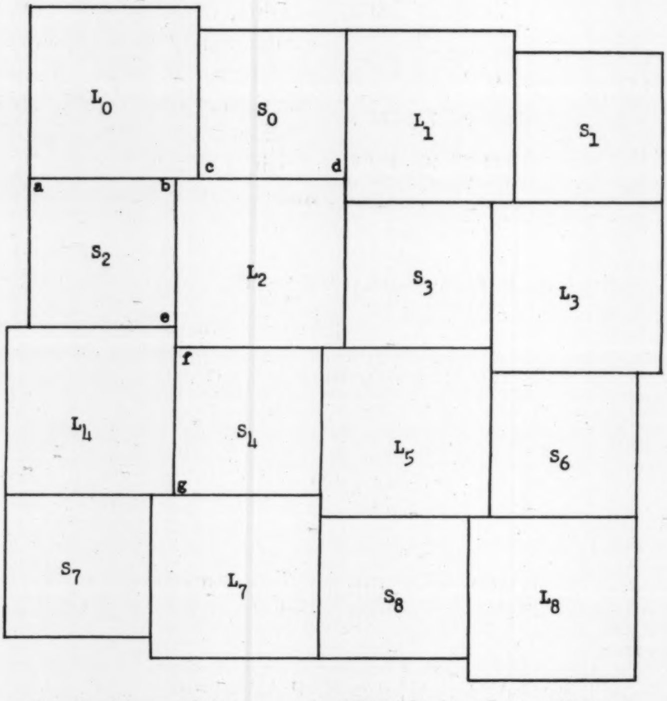

FIG. 1.—Suggested Arrangement of Particles in a Cement Paste.

being formed, in the reaction except the small number in parentheses in the right-hand side of each reaction. This is the number of cubic centimeters by which the volumes of the reactants exceed the volumes of the reaction products. These calculations show that the reaction products in all cases occupy less volume than the sum of the volumes of the reactants. This means that if the reactants dissolved in the liquid phase of the cement paste and then precipitated as

The second line of numbers under the equation shows the ratio of the volume of the principal reactant to the volume of the solids produced by the reaction. For example, 1 volume of tricalcium aluminate produces 8.0 volumes of calcium sulfoaluminate by the reaction of Eq 1. On the other hand, Eq 5 shows that 1 volume of tricalcium silicate produces only 1.55 volumes of tobermorite and $Ca(OH)_2$.

When I proposed this solid state reaction theory, I pointed out that it would explain why portland-cement mortars and concretes continued to expand when stored under water. The theory has been opposed on the basis that reactions of that type would produce relatively enormous expansions during setting and hardening. One does not know in detail how the particles of cement are arranged with respect to each other in pastes. However, one can learn something by studying an idealized arrangement. On the assumption that the cement particles are cubes of one size and that the paste consists of 40 parts cement and 60 parts water by volume, the water can be visualized as being present in cubes 1.15 times larger than the cubes of cement. That is the ratio of 40 to 60 is 1.0 to 1.5 and the cube roots of 1.0 and 1.5 are 1.0 and 1.15, respectively. Figure 1 is a schematic representation of a plane through such a paste in which the S units are solids and the L units are liquid. The lines *ab*, *cd*, etc., are 1 unit long and the lines *bc*, *ef*, etc., are 0.15 units long. This figure shows each grain to be separated from the others by layers of water. In this particular paste, the average thickness of the layers of liquid at the points at which the particles are closest to each other is 0.15 units. If this figure is realistic, and there appears to be no reason to believe that it is not, then the particles of cement can expand to more than double their original volume without interference from a neighboring particle except at certain points of closest approach. This is, when the edge *ab* of the cube S_2 expands, it will tend to expand into the space represented by the line *bc*, and the edge *cd* of the cube S_0 will also tend to expand into this space.

It is seen from Eq 5, that a crystal of tricalcium silicate when fully hydrated to tobermorite and calcium hydroxide expands 55 per cent, or 1 volume expands to a volume of 1.55. The cube root of 1.55 is 1.16. Suppose the cubes S_0 and S_2 of Fig. 1 are tricalcium silicate. When they hydrate, the edges *cd* and *ab* would tend to expand equally in both directions. They would each tend to expand 0.08 units into the space represented by the line *bc*. Hence, the two cubes are tending to expand 0.16 units into a space of 0.15 units. Each cube had an original length of 1 unit, and each has now expanded to a length of 1.16 units. The length available for the expansion of each cube is the original 1.00 unit of the solid plus the 0.15 units of water-filled space, and $\frac{1.16 - 1.15}{1.15} \times 100 = 0.87$. From this it appears that the tricalcium silicate could produce less than 1.0 per cent expansion in this paste, if it were transformed directly to tobermorite and calcium hydroxide by the reaction of Eq 5. Dicalcium silicate would produce less expansion. This degree of interference and expansion would occur only at certain points where the grains approached each other rather closely and when the crystals were completely hydrated.

When tricalcium aluminate chemisorbs calcium sulfate and water to form the calcium sulfoaluminates or the hydrated tetracalcium aluminate, the crystals expand several hundred per cent. Since the crystals of tricalcium aluminate should be uniformly distributed among the other crystals, there should be water-filled space surrounding each crystal sufficient to accommodate most of the expansion except at the points where they are close to other crystals. As the space represented by the lines *bc*, *ef*, etc., in Fig. 1 becomes filled with reaction products, the paste becomes rigid. According to the data presented by Powers, some pastes develop sufficient rigidity within the first two hours to show dilation from these reactions of hydration. It seems very likely that most of the expansion that occurs during the early periods is produced by the reactions of tricalcium aluminate with either calcium sulfate or calcium hydroxide, or with both.

Returning now to the statement by Mr. Kennedy, it appears that that statement is incorrect with respect to the sulfoaluminates occupying more volume than the sum of the volumes of the reactants when water is considered as a reactant. Also it appears that the formation of sulfoaluminate in the paste causes some expansion during the first 24 hr. However, experience seems to show that any expansion that does occur in the first 24 hr is beneficial with respect to strength and drying shrinkage.

Lerch (5) showed that as the calcium sulfate content of a cement was increased to some optimum amount, the strengths increased, and the drying shrinkage decreased.

It is seen from the above calculations that when the water content of the paste is not changed, the effect of added sulfate should be to fill more of the original water-filled space. One would expect this to result in increased strengths and decreased drying shrinkage. However, if the added gysum was sufficient to disrupt the mass as the cement minerals were hydrating, the effect might be to produce unfilled space in the form of minute cracks that would weaken strength and increase drying shrinkage.

If hydrated tricalcium aluminate, as represented by Eq 4, as well as anhydrous tricalcium aluminate can chemisorb calcium sulfate and water to be directly converted into calcium sulfoaluminate, one can understand why products produced from cements of relatively high tricalcium aluminate contents expand when exposed to sulfate solutions. On the other hand, one would not expect expansions if the aluminates dissolved in the sulfate solution and then crystallized as the sulfoaluminate.

The aluminoferrites appear to form sulfoaluminates during the hydration of the cement because cements that theoretically contain no tricalcium aluminate combine with calcium sulfate in pastes as they set and harden. However, the aluminoferrites do not appear to cause expansion in sulfate solutions. Since these compounds are dibasic compounds, they may not be able to chemisorb calcium sulfate and water for a direct transformation to a solid sulfoaluminoferrite. In an excess of water these compounds appear to react with calcium sulfate to give a calcium aluminosulfate and hydrated ferricoxide. This probably is a through solution reaction and it may be that in cement pastes that are partially hydrated the water content is too low to cause this reaction to proceed at an appreciable rate. This seems to be one of the unsolved problems in the chemistry of the hydration of cement.

MR. THOMAS B. KENNEDY (*author*).—The expansion, cracking, and general disintegration occurring as the result of reaction between tricalcium aluminate and calcium sulfate or between tricalcium aluminate and the sulfate ion from a different source in the presence of calcium hydroxide has led many of us to the apparently erroneous conclusion that the volume of the reaction products is greater than the sum of the volume of its components. We are accustomed to thinking in the terms of through solution reactions.

I am greatly indebted to Mr. Hansen for his lucid discussion of the reactions and his explanation of the solid state mechanism that causes expansion.

Mr. Hansen's explanation of why the addition of the correct amount of calcium sulfate to cement has a beneficial effect on strength and drying shrinkage is also most appropriate.

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SIGNIFICANCE OF TESTS FOR SULFATE RESISTANCE*

BY WILLIAM LERCH[1]

It has been recognized for many years that certain characteristics of cements play an important part in the sulfate resistance of concrete. The areas of so-called "alkali-soils" are sufficiently widespread to justify consideration of sulfate resistance in the specifications for portland cement. This problem has been studied by many investigators during the past 60 years (**1**)[2]. About 1935 the ASTM Committee C-1 on Cement appointed a working committee to study the problem. In 1940, Advisory Committee for the Long-Time Study of Cement Performance in Concrete considered the problem of sufficient importance to include, in that study, a project in which concretes made from different cements were exposed to alkali soil water under field conditions (**2**).

CHEMICAL LIMITATIONS

Chemical limitations provide a basis for specifying portland cements that will provide good quality concrete with excellent sulfate resistance. It is generally recognized that the C_3A content of the cement has a significant effect on sulfate resistance, but it is recognized also that there are some factors other than C_3A that influence the sulfate resistance. For example, it was concluded from the Long-Time Study of Cement Performance in Concrete (**2**) that:

> While no quantitative relationship between the resistance of the concrete to sulfate attack and C_3A content of the cement was exhibited in these tests, the relationship was such that the percentage of C_3A provides a fairly good criterion of sulfate resistance. The fact that there are several striking exceptions to the relation between sulfate resistance and the C_3A content indicates that there is some unrecognized factor in the cement composition, or in the manufacture of the cements, which affects the sulfate resistance.

The problem of sulfate resistance is

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* Presented at the Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

recognized in the ASTM Standard Specifications for Portland Cement (C 150)[3] and the Standard Specifications for Air-Entraining Portland Cement (C 175)[3]. In these specifications, the sulfate-resistant cements are defined in terms of chemical limitations. Type II and IIA cements are recommended for use in general construction exposed to moderate sulfate action and type V cement for use when high sulfate resistance is required. The chemical limitations in the current specifications appear to be adequate for the selection of portland cements that will produce concrete having a high resistance to sulfate waters. Good quality concretes made with types II, IIA, and V cements have given excellent performance under adverse conditions of alkaline sulfate exposure. However, such chemical limitations are not equally applicable in specifications for portland blast-furnace slag cements or portland-pozzolan cements. It would be desirable to have a performance test that would be suitable for all types of cement.

PERFORMANCE TESTS

The Working Committee on Sulfate Resistance has pursued the development of a performance test for the past 20 years. The earlier studies were directed to measuring the expansion of lean mortar bars immersed in sulfate solutions and the evaluation of a sulfate susceptibility test. The results of these studies have been published in two reports (3, 4). Each of these methods of test did have considerable merit. However, the degree of reproducibility of results obtained by different laboratories was not considered by Committee C-1 to be sufficiently satisfactory to justify the recommendation that either method should be adopted as an ASTM method of test for sulfate resistance.

In 1956 the Working Committee on Sulfate Resistance reported (5) on studies of a new procedure for determining the potential sulfate resistance of portland cements. The method of test consists of measuring the expansion of 1:2.75 graded Ottawa sand mortar prisms containing excess calcium sulfate (gypsum) equivalent to a total SO_3 content of 7.0 per cent by weight of the original cement plus added gypsum. The mortar prisms were cured in the molds one day and then stored in water at 23 ± 1.7 C (73.4 ± 3 F). The percentage expansion of the mortar prisms was calculated and recorded as the per cent change in length on the basis of the length at one day.

A cooperative series of tests was conducted to determine the reproducibility of this method. Thirteen laboratories participated in the testing of 17 cements. The data reported from these tests (5) led to the adoption of the test as an ASTM Tentative Method of Test for Potential Sulfate Resistance of Portland Cement (C 452 - 60 T).[4]

RELATIONSHIP BETWEEN EXPANSION OF MORTAR SPECIMENS AND FIELD PERFORMANCE OF CONCRETES

The concretes exposed to sulfate soils at Sacramento, Calif., as part of the Long-Time Study of Cement Performance in Concrete (2) provided an excellent opportunity to study the relationship between the expansion of mortar specimens with excess gypsum and the field performance of concretes. Twenty-seven cements were used in concretes exposed to sulfate soils in 1942. The cements comprised one or more of each of the five types of portland cements and six air-entraining cements. The complete chemical analyses of these

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[3] 1958 Book of ASTM Standards, Part 4.

[4] 1960 Supplement to Book of ASTM Standards, Part 4.

TABLE I.—C_3A CONTENT OF CEMENT, EXPANSION OF MORTAR PRISMS, AND SULFATE RESISTANCE RATING OF CONCRETES.

Cement		Expansion of Mortar Prisms at 28 Days, per cent	Rating of Concrete Beams[a]							
No.	C_3A Content, per cent		Basin I				Basin II			
			Cement Content, sack per cu yd				Cement Content, sack per cu yd			
			4.0	5.5	7.0	Avg	4.0	5.5	7.0	Avg
PORTLAND CEMENTS										
24	4.4	0.030	5.1	2.7	1.3	3.0	3.7	2.5	1.7	2.6
51	2.2	0.033	4.2	2.1	1.3	2.5	3.5	2.0	1.5	2.3
23	2.1	0.038	4.7	3.0	1.2	3.0	3.0	2.1	1.6	2.2
42	2.7	0.040	3.8	2.5	1.4	2.6	3.6	2.3	1.6	2.5
41	3.4	0.042	4.2	2.8	1.4	2.8	3.7	2.4	1.6	2.6
34	5.2	0.043	5.2	3.0	2.7	3.6	3.0	2.6	2.5	2.7
43A	3.9	0.049	4.1	2.1	1.2	2.5	3.6	2.3	1.6	2.5
25	3.8	0.052	5.2	3.1	1.2	3.2	3.8	2.5	1.5	2.6
21	5.1	0.052	4.8	2.9	1.4	3.0	4.0	2.3	1.8	2.7
14	7.1	0.054	5.4	3.7	2.2	3.8	4.6	3.6	2.0	3.4
13	8.9	0.060	5.4	4.6	2.0	4.0	4.8	3.7	2.2	3.6
16	6.4	0.062	5.2	3.9	3.1	4.1	3.9	3.2	2.7	3.3
22	5.4	0.066	5.3	4.4	1.6	3.8	4.6	3.3	2.1	3.3
43	4.8	0.068	4.1	2.7	1.3	2.7	3.2	2.3	1.6	2.4
17	9.1	0.072	5.4	4.2	3.2	4.3	4.6	3.7	3.0	3.8
31	9.6	0.092	5.7	3.6	3.3	4.2	4.8	3.7	3.3	4.1
33	9.2	0.093	5.9	4.0	3.1	4.3	4.9	3.4	3.2	3.8
11	10.9	0.102	5.8	4.7	2.1	4.2	5.4	4.7	3.1	4.4
15	9.9	0.117	5.9	4.7	3.4	4.3	5.4	4.7	2.7	4.3
12	11.2	0.135	5.6	4.9	3.5	4.7	5.3	4.7	3.1	4.4
18	12.2	0.224	5.8	4.9	4.1	4.9	5.5	5.1	4.0	4.9
AIR-ENTRAINING PORTLAND CEMENT										
21T	5.4	0.065	3.6	1.4	1.2	2.1	3.6	1.8	1.6	2.3
16T	6.8	0.065	3.9	1.3	1.3	2.2	3.1	2.1	1.5	2.2
33T	9.3	0.116	4.9	3.2	2.0	3.4	4.1	2.8	2.2	3.0
11T	11.0	0.123	5.1	4.2	1.6	3.6	4.6	3.7	2.6	3.6
12T	11.1	0.166	5.4	4.4	3.4	4.4	5.2	4.7	3.5	4.5
18T	12.2	0.225	5.2	4.5	3.9	4.5	5.4	4.9	3.8	4.7

[a] Numerical ratings range from 1 for unaffected specimens to 6 for those completely deteriorated.

cements have been shown in a previous report (**5**).

Three concrete mixes were used for each of the 27 cements. These mixes were designed to have the following cement contents and consistencies within the practical limits of control: mix 1 had 4 sacks of cement per cu yd with a $1\frac{1}{2}$-in. slump; mix 2 had 5.5 sacks of cement per cu yd with a $1\frac{1}{2}$-in. slump; and mix 3 had 7 sacks of cement per cubic yard with a $1\frac{1}{2}$-in. slump. An equal number of specimens from each mix were placed in each of two different basins containing natural and prepared sulfate soils. For basin I, natural soil from Willows, Calif., which is high in sodium sulfate and low in magnesium

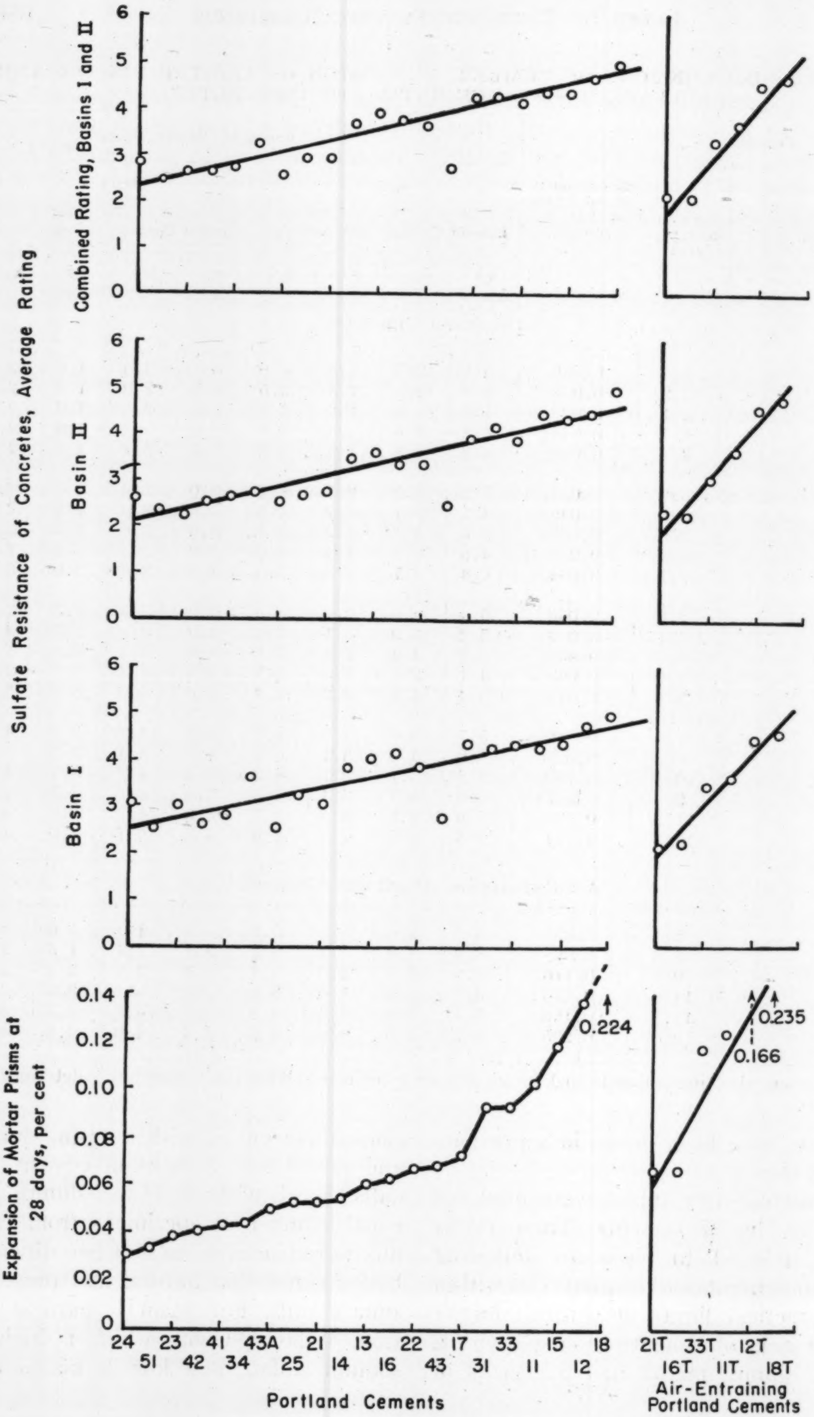

FIG. 1.—Expansion of Mortar Prisms Versus Sulfate Resistance of Concretes in Order of Ascending Expansion of the Mortar Bars.

sulfate was used. For basin II, a soil of higher magnesium sulfate content was used.

The specimens were rated on the basis of visible evidence of disintegration. By this procedure, numerical ratings were assigned ranging from one for unaffected specimens to six for those completely deteriorated. The specimens were inspected and rated three times a year for the first two years and once a year thereafter. A numerical value for sulfate resistance of concrete made with any one cement was obtained by using the average cumulative value of the ratings of specimens made with the cement. The ratings reported at each inspection for one concrete mix in one basin, were added and the sum divided by the number of ratings which provided a rating for the cement in that particular mix for that basin. The average of the ratings for the three different mixes provided a grand average numerical value for the sulfate resistance of the cement in that basin.

The 27 cements used in the field exposure tests of concretes exposed to sulfate soils were tested in mortar prisms with excess gypsum by the method described in ASTM Method C 452 - 60 T.[4] Table I shows the expansion of the mortar bar specimens at the age of 28 days, the sulfate resistance of the concretes, and the C_3A content of the cements. In this table, the cements are arranged in the order of ascending expansion of the mortar prisms. Many of the lean-mix concretes, 4.0 and 5.5 sacks per cu yd, disintegrated rapidly in basin I. For that reason, the ratings for those two mixes in basin I are for 6 yr exposure in order to provide better discrimination between the different cements. The remaining ratings shown in Table I are the cumulative ratings of the concretes after 18 yr exposure to sulfate soils.

The data in Table I show that as the expansions of the mortar prisms increase, the sulfate resistance of the concretes decreases. Figure 1 provides a further comparison of the relationship between the expansion of the mortar prisms and the sulfate resistance of the concretes. The cements are arranged in the order of ascending expansion of the mortar prisms at 28 days as shown in the lower diagram. The two middle diagrams show the sulfate resistance rating of concrete specimens exposed to different soils, basins I and II, respectively. The top diagram shows the combined rating of the concretes, that is, the average for basins I and II. These diagrams show a relatively good relationship between the 28-day expansion of the mortar prisms and the sulfate resistance of the concretes. It will be noted that the original LTS series included only one type V cement. In 1949, thirteen additional cements were used in the preparation of concrete specimens for exposure to sulfate soils. The cements were of types II, IV, and V and they were used in concrete mixtures containing 5.5 and 7.0 sacks of cement per cu yd. In 1958, three portland blast-furnace slag cements and portland cements made from the same clinker were used in the preparation of concrete specimens for exposure to sulfate soils. All of these cements have been tested in mortar prisms with excess gypsum as described in ASTM Method C 452 - 60 T. However, the concretes made with these cements have shown very little deterioration to date, so their sulfate resistance cannot be related to the laboratory test at this time. That will be done at a later date.

There are no data available at this time to show the relationship between the expansion of mortar prisms containing excess gypsum and the sulfate resistance of concretes when using portland blast-furnace slag cements or portland-pozzolan cement. Mather (6) has reported that the expansion of mortar prisms made with portland blast-furnace slag cements with excess gypsum was approximately the same as that of similar specimens made with portland cements having the same C_3A content as the clinker component in the portland blast-furnace slag cement. However, when portland cements and blends of portland cement with pulverized blast-furnace slag were used in the preparation of lean-mortar bars for immersion in a 5 per cent solution of sodium sulfate, the bars, made with the blends had much better sulfate resistance than those made with the portland cements. It was suggested that the test using excess gypsum does not allow the slag constituent of portland blast-furnace slag cements to manifest its effect in promoting sulfate resistance.

TABLE II.—SULFATE RESISTANCE OF TYPE I PORTLAND CEMENT AND TYPE IS PORTLAND BLAST-FURNACE SLAG CEMENT.

Cement		C_3A Content of Portland Cement or Clinker, per cent	Mortar Prisms with Excess Gypsum, ASTM C 452 - 60 T					Lean-Mortar Prisms Immersed in 5 per cent Na_2SO_4 solution				
			Expansion, per cent					Expansion, per cent				
Lot No.	Type		7 days	28 days	3 mo	6 mo	1 yr	1 mo	2 mo	3 mo	4 mo	5 mo
19462...	I	11.6	0.064	0.141	0.332	0.854	0.894	0.025	0.055	0.163	(at 11 weeks)	
19461...	IS	11.6	0.076	0.154	0.161	0.164	0.169	0.020	0.023	0.026	0.029	0.030
19465...	I	12.8	0.064	0.171	0.790	0.804	0.812	0.112	(Badly warped after 1 mo)			
19464...	IS	12.8	0.065	0.179	0.422	0.426	0.429	0.028	0.212	(Disc. at 2 mo)		
19468...	I	9.1	0.041	0.091	0.191	0.313	0.409	0.023	0.030	0.056	0.120	Disc.
19469...	IS	9.1	0.043	0.109	0.313	0.468	0.470	0.018	0.022	0.027	0.034	0.042

Klieger and Isberner (7) have made laboratory tests for sulfate resistance of type I portland cements and type IS portland blast-furnace slag cements made from the same clinker. They measured the expansion of mortar prisms with excess gypsum as described in ASTM Method C 452 - 60 T, and the expansion of lean-mortar prisms immersed in a 5 per cent sodium sulfate solution as described in Appendix A of the Report of Committee C-1 on Cement (8). The results of their tests are shown in Table II. The expansions at 28 days of the mortar prisms with excess gypsum are essentially the same for the portland cements and the portland blast-furnace slag cements made from the same clinker. However, for the lean-mortar bars immersed in the sodium sulfate solution, the portland blast-furnace slag cements show a significantly higher sulfate resistance than do the comparable portland cements made from the same clinker.

The data now available seem to indicate that the mortar prism test with excess gypsum, ASTM Method C 452 - 60 T, is not an adequate performance test for portland blast-furnace slag cements. Additional tests now under way, including concretes exposed to sulfate soils, will provide further information on this subject.

CONCLUSION

Progress has been made in the development of a performance test for sulfate resistance. The ASTM Tentative

Method of Test for Potential Sulfate Resistance of Portland Cement (C 452 - 60 T) appears to be an acceptable performance test for the sulfate resistance of portland cements. Data now available seem to indicate that the test is not suitable for use with portland blast-furnace slag cements. Additional tests now under way will provide further information on that subject.

It would be desirable to have a performance test for sulfate resistance that would be suitable for use with all types of cement. There is a particular need for such a performance test for portland blast-furnace slag cements and portland-pozzolan cements. Work on the development of a performance test for sulfate resistance is being continued.

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DISCUSSION

MR. T. THORVALDSON[1] (*presented in written form*).—ASTM Committee C-1 on Cement is to be commended for its continuing efforts to develop a simple test for cement, suitable for predicting the performance of concretes exposed to sulfate water under field conditions. It is good news that ASTM Method C 452 - 60 T "appears to be an acceptable performance test for the sulfate resistance of portland cements."

Methods involving the expansion of lean mortars in sulfate solutions, in addition to presenting problems of reproducibility, fail due to the fact that they exclude protective factors which come into prominent action only in mass concrete of fairly high cement content. Thus while the relative rates of expansion of very lean cement mortars may indicate the relative chemical reactivity of the cements, they would not indicate the relative resistance to sulfate action of concretes of high cement content unless composition of the cement changes materially, or if the physical properties of the concrete are altered, for example, through addition of "active" additives.

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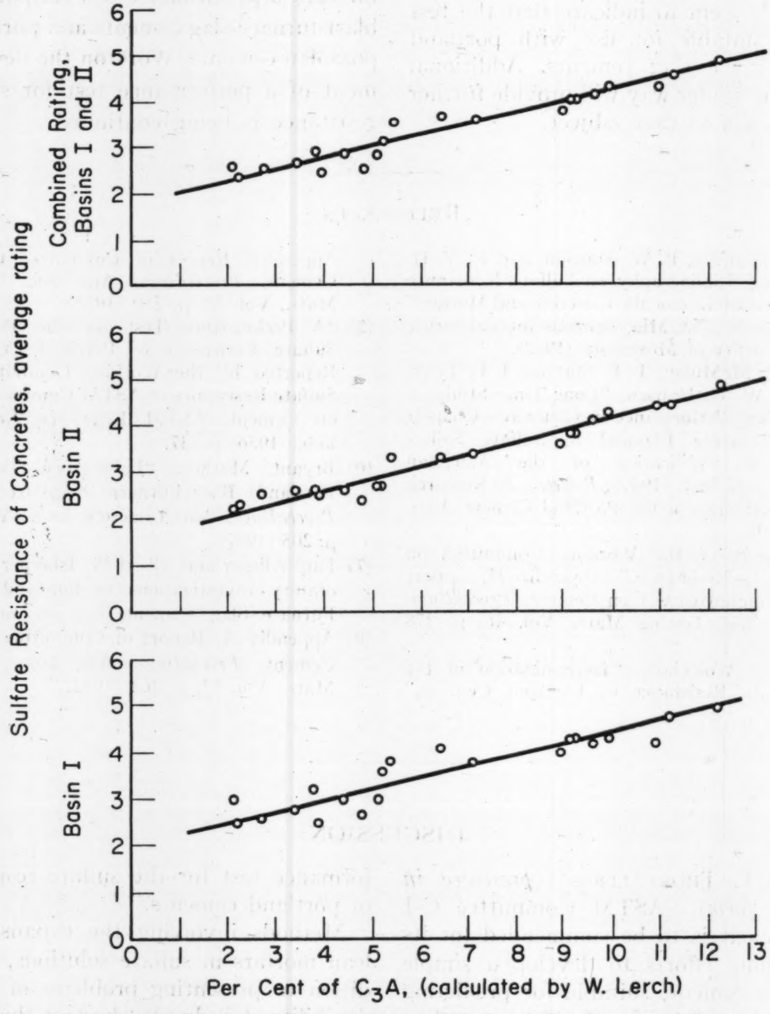

FIG. 2.—Sulfate Resistance of Concrete Versus Calculated C_3A Content of Cement.

these protective factors, in the case of the cements compared, were to develop at the same constant rate as the cement content is increased. Such coincidence does not appear to be likely if chemical This seems to militate against developing a single simple test for all types of concrete.

Mr. Lerch has attempted to present the experimental data in Fig. 1 in a completely objective manner using the expansions obtained under ASTM Method C 452 - 60 T only for the purpose of arranging the cements in ascending order of expansion at 28 days. The ratings of the concretes obtained at Sacramento are then plotted against a "unitary" scale of individual cements, irrespective of the increases in expansion of the mortar from cement to cement. The picture would be somewhat changed if the numerical ratings of each concrete were plotted against the expansion of the mortar obtained for each cement.

The writer has plotted the Sacramento ratings for concretes against the C_3A content of the cements as given in the report (see the accompanying Fig. 2). The rating points, allowing for the change in the scale, are on the whole closer to the average straight line than in Fig. 1 of the paper, and the largest variations have disappeared or been reduced.

There are many reasons, theoretical and practical, why the ratings versus calculated C_3A plot might be expected to give erratic results. In making the calculation of per cent C_3A, it is assumed that the Fe_2O_3 is present as C_4AF and that all the excess Al_2O_3 is present as a C_3A phase. But the "iron" phase may contain Al_2O_3 as C_6A_2F in solid solution. The amount of the iron phase and the amount of "glass" it contains is known to modify the sulfate resistance of the concrete materially. According to the data given in reference (2) of the paper, the calculated C_4AF in the 21 cements varies from 6.5 to 16.6 per cent and bears no relation to the calculated per cent of C_3A present, thus imposing a variable factor for each concrete. Similarly other factors affecting sulfate resistance may be introduced by the minor oxide components of the cement and by variations in the specific surface.

The result obtained by plotting the ratings for the sulfate resistance of the concretes exposed in the Sacramento "basins" against the very hypothetical calculation of C_3A in the cements is, therefore, quite striking and supports the idea of the overwhelming importance of the Al_2O_3 and Fe_2O_3 content of a portland cement on the resistance to sulfate action of a well-made concrete.

FLEXURAL STRENGTH OF HYDRAULIC CEMENT MORTARS*

BY MYRON A. SWAYZE[1]

The testing of mortar prisms for flexural strength is supposed to have originated in France in 1904, according to information from Swedish authorities, one of whom used the method in Zurich, Switzerland, in 1906, "to prove that bending strengths were twice the tensile strength of briquets." After these original tests the method lay dormant for a considerable number of years. The Swiss adopted the method in their standard specifications in 1933. A similar method was used in Germany to test cements for use in their Autobahnen beginning in 1934. The test was made part of the German specifications for all cements in 1939, and was in general use by 1942. Sweden adopted the method in their specifications in 1943, with Finland following suit at about the same time. A number of other European countries are now seriously considering the method.

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ASTM Tentative Methods C 348[2] and C 349[3] on the flexural and compressive strengths of 2 by 2 by 12-in. prisms were issued in 1954. Since each of these large prisms required a full batch of C 109[4] mortar, the tests had little chance of growing in favor. The C-1 Working Committee on Strength revised these methods in 1957, to reduce specimen size to the 40 by 40 by 160-mm prism now in use in Europe. This change permits molding of three specimens from each standard batch of C 109 mortar. Span lengths for breaking prisms in flexure and bearing area for modified cube compression tests were revised slightly from European dimensions to convert breaking loads into pounds per square inch by use of simple conversion factors.

In the Lone Star Cement Research Laboratory at Hudson N. Y. experience with molding these 40-mm prisms over the past seven years and with breaking them in flexure and as modified cubes for compressive strength, has shown a high degree of uniformity among specimens as cast from the same cements and broken at the same ages, as compared with similar tests on briquets. Coefficients of variation among 9 specimens for each cement in a large group of type I, II, III, IA, and IIA cements have averaged as follows:

Type of Test	Coefficient of Variation
Briquets	4.41
2-in. cubes	2.50
Prisms, flexure	2.35
Prisms, compression	2.39

During the period 1957–1960, the Lone Star Cement Research Laboratory ran a comprehensive series of tests on 56 cements of all types except IV and V. Included in these tests were concrete tests in flexure and compression with cement contents of 3.6, 4.5, 6.0, 7.5, and 9.0 sacks per cu. yd., with water additions to yield 2-in. and 6-in. slumps. Standard briquets, 2-in. cubes, and 40-mm prisms were also made on each cement. Three rounds of tests were made for each type of test specimens and test age. The concrete tests were made on 6 by 6 by 30-in. beams and 6 by 12-in. cylinders. All concrete mixes were made with non-reactive siliceous sand and gravel graded from 0 to $1\frac{1}{2}$ in. All non-air-entraining batches were designed to have a total fineness modulus of 4.85, with cement volume included with an assigned fineness modulus of 0.00. Air entraining concrete batches were de-

TABLE I.—RATIO OF COMPRESSIVE TO FLEXURAL STRENGTHS IN CONCRETE AND IN STANDARD MORTAR PRISMS.

Test Age	Types I and II	Types IA and IIA	Type III	Type IIIA
RATIO OF CONCRETE COMPRESSIVE STRENGTH TO CONCRETE FLEXURAL STRENGTH				
1 day	3.75	3.77	4.92	5.23
3 days	4.89	4.77	5.92	5.82
7 days	5.44	5.42	6.20	6.20
28 days	6.23	6.20	6.85	6.62
RATIO OF PRISM COMPRESSIVE STRENGTH TO PRISM FLEXURAL STRENGTH				
1 day	3.94	3.62	4.73	3.86
3 days	4.38	4.22	5.77	4.97
7 days	5.09	4.79	6.04	5.65
28 days	5.74	5.30	6.52	5.98

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[2] ASTM Method of Test for Flexural Strength of Hydraulic Cement Mortars (Tentative) (C 348 – 57 T), 1958 Book of ASTM Standards, Part 4.

[3] ASTM Method of Test for Compressive Strength of Hydraulic Cement Mortars (Using Portions of Prisms Broken in Flexure) (Tentative) (C 349 – 59 T), 1959 Supplement to Book of ASTM Standards, Part 4.

[4] ASTM Method of Test for Compressive Strength of Hydraulic Cement Mortars (Using 2 in. Cube Specimens) (C 109 – 58 T), 1958 Book of ASTM Standards, Part 4.

signed to have a total fineness modulus of 5.00.

For purposes of this report, concrete flexural and compressive strengths for cement contents of 4.5, 6.0, and 7.5 sacks per cu yd and slumps of 2 and 6 in. were averaged for each cement, yielding individual values for each test age on an average of 18 specimens for each cement. Single values on mortars for each cement and test age were averages of 9 specimens of briquets, 2-in cubes, and prisms in flexure and of 18 tests on prisms broken as modified cubes. Table V of this report shows grand averages for all tests on all cements in the same strength range group.

From the data in this table, a considerable number of comparisons of different kinds of strength tests are available. Less importance should be attached to the data on type IIIA cements than to the data on other groups, since only four of these cements were tested.

It has long been recognized that the ratio of flexural and compressive strengths of concrete is not a constant for any particular age of test but varies with different types of coarse aggregate. Rounded gravels yield lower modulus of rupture strengths for a given compressive strength than do crushed materials. Methods of testing in flexure also yield different values, third-point loading giving lower strengths than center-point loading. In the present tests, both mortar prisms and concrete beams were broken by center-point loading.

Likewise, the relative proportions of flexural and compressive strengths will vary with degree of hydration of cement in the specimens. Since for any type of mix, compressive strengths develop faster than flexural strengths, we should find an increasing ratio between the two types of test with age. Type III and IIIA cements hydrate materially faster than Type I or II cements or their air-entraining counterparts, and we should, therefore, expect to find their compressive - flexural strength relations farther advanced at a given age than for cements of normal fineness and composition. Finally, the quantity of entrained air in parallel specimens must have an influence on compressive - flexural strength relations. The effect of air entrainment on this relation is much greater in standard mortar specimens than in concrete due to the much greater quantities of air in mortars.

TABLE II.—RATIO OF FLEXURAL STRENGTH TO FLEXURAL STRENGTH OF PRISMS AND TENSILE STRENGTH OF BRIQUETS.

Test Age	Types I and II	Types IA and IIA	Type III	Type IIIA
RATIO OF CONCRETE FLEXURAL STRENGTH TO FLEXURAL STRENGTH OF PRISMS				
1 day	1.17	1.27	0.85	0.78
3 days	0.96	1.04	0.78	0.80
7 days	0.85	0.89	0.73	0.77
28 days	0.79	0.80	0.73	0.78
RATIO OF CONCRETE FLEXURAL STRENGTH TO TENSILE STRENGTH BRIQUETS				
1 day	1.95	2.16	1.65	1.44
3 days	1.71	1.76	1.54	1.49
7 days	1.57	1.61	1.50	1.50
28 days	1.65	1.57	1.53	1.50

Table I shows relations of compressive strength to flexural strengths in concrete and in standard mortar prisms at the various test ages.

It can be seen from these data that the hydration of type III and IIIA cements in concrete at 1 day is about equal to that of normal cements at 3 days, while their 7-day values approximate 28-day ratios for normal cements closely. About the same ratios hold for the strength relations in mortar prisms, although here the values are more affected by the large increases in air as we go from normal to air-entraining varieties of the cements.

For predicting the flexural strengths of nonair-entraining concrete made with the particular coarse aggregates used in our tests, there appears to be little choice between the flexural strength of mortar prisms and the tensile strength of the old-fashioned briquet. The notable difference in the two types of strength relations shown in Table II is that the concrete briquet strength ratios turned upward after the age of 7 days, due to brittleness and high internal stresses in the briquet as breaks occur in older specimens.

TABLE III.—RATIO OF COMPRESSIVE STRENGTH TO TENSILE STRENGTH OF BRIQUETS AND FLEXURAL AND COMPRESSIVE STRENGTHS OF PRISMS.

Age	Types I and II	Types IA and IIA	Type III	Type IIIA
RATIO OF CONCRETE COMPRESSIVE STRENGTH TO BRIQUET TENSILE STRENGTH				
1 day	7.32	8.14	8.09	7.54
3 days	8.35	8.42	9.12	8.67
7 days	8.50	8.70	9.32	9.28
28 days	10.28	9.73	10.48	9.93
RATIO OF CONCRETE COMPRESSIVE STRENGTH TO PRISM FLEXURAL STRENGTH				
1 day	4.38	4.80	4.19	4.11
3 days	4.71	4.98	4.62	4.64
7 days	4.64	4.80	4.51	4.78
28 days	4.91	4.97	4.97	5.17
RATIO OF CONCRETE COMPRESSIVE STRENGTH TO PRISM COMPRESSIVE STRENGTH				
1 day	1.11	1.33	0.89	1.06
3 days	1.05	1.18	0.80	0.94
7 days	0.91	1.01	0.75	0.85
28 days	0.86	0.94	0.76	0.86
Water-Cement Ratios	6.10 to 5.43	5.61 to 5.09	6.12 to 5.38	5.78 to 5.16

While briquets were also made for all air-entraining cements in our study, the water content of these mixes as called for by normal consistency tests produced a very sloppy mortar which could not be molded by standard methods. This probably accounts for the improvement in 28-day briquet behavior, since the wetter mortars should have less internal stress at time of rupture and thus yield relatively higher strengths.

On the other hand, the mortar prisms developed no such internal stresses during loading. The gradually descending ratios of concrete flexural strength to prism strength with age can all be attributed to the lower water-cement ratios used in the mortars from which prisms were molded, in comparison with that of the concrete.

The almost complete agreement between ability of the prism flexural test and the briquet to predict the flexural strength of concrete made with type III cement of various test ages should be of much interest to the advocates of the briquet as a primary method of test. Their expressed fears that the transition from the briquet to flexural testing of prisms would remove means of detecting low 24-hr. flexural strengths in high-early-strength concrete are shown to be completely unfounded by the present data. While the ratios of the two tests to concrete flexural strengths are necessarily different, the decline of these ratios with age of specimens are almost exactly in the same proportions. The upturn of the concrete-briquet ratio at 28 days again is due to high internal stresses in type III cement briquets at this age, which causes subnormal strengths. The flexure test in prisms does not have this fault.

While the title of this paper theoretically limits the author to discussion of flexural strengths of cement mortar specimens, practical considerations require that some consideration be given to the utilization of broken prism halves in determining compressive strength on the same mortars. Prior to the publication of Tentative Methods C 348[2] and C 349[3] by the Society, no means were available for testing cements for both tensile and compressive strength at the same realistic water-cement ratio. The present 40-mm prism fills this need. To scrap halves of prisms after making the flexural tests would be wasteful of both materials and testing effort. Relations of the tensile strength briquets and the flexural and compressive strength of prisms to the compressive strength of concrete as shown in Table III should therefore be considered.

Considering first the relations of concrete compressive strength to briquet strengths for the nonair-entraining types of cement—the only cements on which we have briquet strength limits—we should note two points in the data on these relations. First, the wide differences in ratios of compressive strength to tensile strength between the 1 and 28-day tests show that increases in briquet strength lag far behind gains in compressive strength as specimens increase in age. Second, if we assume that these varying strength ratios are normal for types I and II cements, then briquet strengths underestimate the compressive strengths of type III cement concretes, especially at the 1, 3, and 7-day periods.

Contrasted to this behavior of briquets, the prism flexural strengths furnish a better index of compressive strength development in concrete made with sand-gravel aggregates. These relations are best for the type IA and IIA cements, where differences between water-cement ratios of mortars and concretes are least (0.52 gals per sack). They are less accurate for type III concretes, where difference between water-cement ratios are greatest (0.74 gals per sack).

These differences in water-cement ratios among concretes and equivalent mortars also confuse the picture when compressive strengths of concrete and those of the C 109 standard mortar in either 2-in. cubes or 40-mm modified cubes are compared. Where the mortar specimens contain materially less water than the equivalent concrete, as in the case of type III cements, the mortar compression tests materially overestimate concrete strengths at all ages. Regardless of this overestimation, however, the consistency of strength relations for type III cements from 1 day to 28-day results was the best of all types studied. For the other type groups, the tendency of the mortar tests was to underestimate early concrete strengths and overestimate the later ones.

It would appear from these studies that the art of testing cements for strength in mortars would be improved if uniform water-cement ratios were used as is done universally in Europe at present and as was once done in the C 109 method for compressive strength of 2-in. cubes. This change would materially improve agreement between laboratories on tests of individual cements and should

TABLE IV.—WATER CONTENTS OF CONCRETES AND SUGGESTED WATER CONTENTS OF C 109 MORTARS.

Types of Cement	Water Used in Tests, per cent		Suggested Water, per cent
	Concretes	C 109 Mortars	C 109 Mortars
I and II	54.1	48.1	50.0
IA and IIA	49.8	45.1	47.5
III	54.2	47.7	50.0
IIIA	51.2	45.8	47.5

also result in better concordance between mortar and concrete tests. Tests for flow on the 10-in. table might be retained to note any unusual departures in consistency, but uniform water-cement ratios should be used.

Some lower water-cement ratio should be adopted for air-entraining cement mortars, since these cements require notably less water in concrete when sand contents are reduced to compensate for

TABLE V.—AVERAGE DATA ON STRENGTHS OF 56 CEMENTS.

	Briquets	2-in. Cubes	40-mm Prisms		Concrete	
	Tension	Compressive	Flexure	Compressive	Flexure	Compressive
25 TYPE I AND II CEMENTS						
Water-cement, gal per sack	4.79	5.43	5.43		6.10	
Strength 1 day	179	1087	299	1179	349	1310
3 days	317	2428	562	2520	541	2647
7 days	420	3727	773	3938	658	3582
28 days	489	5544	1024	5883	806	5027
12 TYPE IA AND IIA CEMENTS						
Water-cement, gal per sack	4.75	5.09	5.09		5.61	
Strength 1 day	172	961	292	1057	372	1402
3 days	315	2152	532	2246	555	2652
7 days	401	3480	727	3478	645	3495
28 days	471	4666	922	4886	739	4583
15 TYPE III CEMENTS						
Water-cement, gal per sack	4.91	5.38	5.38		6.12	
Strength 1 day	325	2746	628	2972	534	2632
3 days	450	4709	888	5127	692	4097
7 days	511	6146	1055	6364	768	4763
28 days	545	7112	1150	7498	834	5709
4 TYPE IIIA CEMENTS						
Water-cement, gal per sack	4.91	5.16	5.16		5.78	
Strength 1 day	312	2100	572	2208	449	2352
3 days	434	3905	810	4021	646	3762
7 days	470	5262	913	5156	704	4360
28 days	512	5749	982	5888	767	5077

the additional entrained air. Converting the water-cement ratios given in Table V from gallons per sack to percentage water by weight of cement, Table IV shows water percentages used in the various types of concrete and C 109 mortars and a suggestion for uniform water contents of C 109 mortars as used in either 2-in. cubes or 40-mm prisms.

Concerning the future of cement testing for strength properties, it seems somewhat unnecessary to continue making two types of strength specimens of different water-cement ratios, especially when one of the tests is not suitable for use with air-entraining cements. Our present 40-mm prism can supplant both the briquet and 2-in. cube with no loss of useful information, and it will be equally applicable for testing of all varieties of hydraulic cements. The same number of test batches are involved, and molding of prisms is simpler and more productive of uniform results than the same operation on briquets or cubes. It seems time for this change in strength testing methods.

DISCUSSION

MR. BAILEY TREMPER[1] (*presented in written form*).—The author's recommendation that tests of cement for strength in mortars be made with uniform water-cement ratios should be given critical scrutiny before adoption. It may appear to be axiomatic that uniform water-cement ratios would improve agreement between laboratories, but is it necessarily true that those laboratories that cannot make good flow tests can make good strength tests? Sufficient data are available to explore this question. Recently our laboratory completed three rounds of strength tests of five type II cements using a constant, predetermined amount of mixing water. Flows varied from 113 to over 140. What significance can be attached to the strength results in terms of performance of the cements in concrete?

Recent tests of eight type II cements in 6-sack concrete required water-cement ratios varying from 0.553 to 0.582 by weight for constant slump. Ottawa sand mortars made to constant flow with these cements required water-cement ratios that yielded a highly significant correlation with the concrete requirements. It would be difficult to justify the use of a constant water-cement ratio in mortar strength tests of these cements.

MR. MYRON A. SWAYZE (*author's closure by letter*).—Mr. Tremper's objection to the use of uniform percentages of water for making mortar strength tests assumes that flow tests of individual cement mortars will be uniform in different laboratories, if made by competent operators. This assumption is not correct. The C-1 Working Committee on Strength in past cooperative tests has experienced great difficulties in obtaining agreement between well qualified laboratories on water requirement of cement samples in C 109 mortars, and hence on uniformity of the strengths that are reported. The causes for these poor agreements are two-fold.

First, the 10-in. flow table described in ASTM Specifications C 230[2] does not have the precision attributed to it by some users. There are some differences in flexibility of the 10-in. surfaces that lead to differences in vibration on each drop. There is also frequent trouble with lubrication, if the table shaft is to fall freely and yet have no oil present between the contact faces of the table and the supporting frame. The presence of even a small drop of oil between these impact surfaces acts as a damper and will yield low flow results. The invention of the Nujol-silica sand flour by the Cement Reference Laboratory many years ago for standardizing flow tables was in direct recognition of the fact that different tables yielded different flow results on the same cement. Use of this Reference Laboratory mixture has only partly overcome this difficulty.

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[1] Supervising Materials and Research Engineer, California Division of Highways, Sacramento, Calif.

[2] Tentative Specifications for Flow Table for Use in Tests of Hydraulic Cement (C 230 – 61 T), 1961 Book of ASTM Standards, Part 4.

The second cause of flow variations in individual cement mortars in different laboratories is attributable to fineness variations in lots of our standard graded Ottawa sand. It is impossible to maintain this sand closer to an exact grading than as specified at present without increasing the sand cost beyond all reason. The present fineness limits in Method C 109 yield low water-cement ratios and hence high strengths when the sand is in the coarse range, and high ratios and low strengths when the sand approaches the low limits of sieve retention. The use of a uniform water-cement ratio for mixing the C 109 mortars would do much to solve the present difficulty.

Many concrete specifications now have top limits on water-cement ratios for different classes of concrete instead of arbitrary proportions of cement, sand, and coarse aggregate, with water additions variable as needed to provide a narrow range of slump. The testing of cements for strength in mortars made to a uniform water-cement ratio would be of more value in estimating concrete strength in cases where the concrete design is on the uniform water-cement basis. The appreciation of these two difficulties in our present C 109 mortar test method was what prompted the author to suggest a return to uniform water-cement ratios in the present standard test for cement strength.

VARIATIONS IN PORTLAND CEMENT—PART 2

BY STANTON WALKER[1] AND D. L. BLOEM[1]

SYNOPSIS

Fourteen different non-air-entraining cements were sampled monthly for one year and the samples tested for compressive strength at ages of 7, 28, and 91 days. For each period, tests were made concurrently with a control cement which had been blended and sealed in advance. All cement samples were also tested for air-entraining capacity. The program was similar to work reported previousy which involved five sources of cement sampled biweekly for one year.

All sources of cement varied in strength from sample to sample. The degree of variability was greater for some than for others and, in many cases, was sufficient to make it difficult to meet concrete strength specifications without uneconomic over design. Air content also varied, but it was substantially always below the 12 per cent maximum specified for non-air-entraining cements.

Tests of multiple samples from various locations in single cars of two cements were highly uniform. Comparisons between two operators showed them to provide virtually identical mortar strength test results, both in magnitude and uniformity. These supplementary data, along with results for the control cement, provided assurance that indicated within-source strength variations were actual and not caused by vagaries in testing.

This paper reports a second investigation of the uniformity of the strength-producing properties of portland cement from individual mills. It supplements an earlier report presented before the 1958 Annual Meeting of the Society.[2] It exemplifies the continuation of a policy to study cement as a material—its quality and, particularly, the uniformity of its quality.

As pointed out in the earlier paper, strength is the basis for acceptance of much concrete. The data presented there demonstrated conclusively that, for standard conditions of test, the strength of concrete is directly related to the strength of standard mortar. It is clear, therefore, that variations in cement have a significant effect on the economical compliance with specifications for concrete strength. Differences in cements from different mills are part of the problem, but their importance is greatly reduced if the level of strength from a given source is maintained within a reasonable range.

The principal objective of this investigation and the earlier one was to develop information on within-mill uniformity as measured by strength. The data should afford guidance as to the over-design necessary in the selection of concrete proportions. Also, it is hoped that the superior uniformity attained by some mills may encourage others to improve their performance.

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[1] Director of Engineering and Associate Director of Engineering, respectively, National Sand and Gravel Assn. and National Ready Mixed Concrete Assn., Washington, D. C.

[2] Stanton Walker and Delmar L. Bloem, "Variations in Portland Cement," *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 1009 (1958).

SCOPE OF INVESTIGATION

Samples of 14 different cements from 13 mills were secured monthly for one year. Ten of the cements were type I and four were type II. One mill furnished both type I and type II. Eleven brand names were represented.

Beginning April, 1960, the 12 successive monthly samples were secured and shipped to the laboratory in sealed waterproofed paperlined cloth bags. They were taken from regular bulk deliveries by 12 ready-mixed concrete producers located in 11 states. A fifteenth cement, used as the control, consisted of a thoroughly mixed blend of five brands from the Washington, D. C., area prepared in advance and stored in sealed metal containers. It was tested concurrently with the samples from outside sources each month.

As promptly as practicable after receipt of the samples for a given month, mortar strength tests were made in accordance with ASTM Standard Method of Test for Strength of Hydraulic Cement Mortars (Using 2-in. Cube Specimens) (C 109).[3] Because of the number of sources, it was not possible to mix mortar with all 14 cements and the control on the same day. For that reason, sources 1 through 7 and sources 8 through 14 were treated as separate groups, with the 7 cements in a group plus the control always being mixed on the same day.

For each sample, the mortar strength tests were made in triplicate on three different days. The two groups were mixed alternately to average out variations in storage time and ambient conditions. Each batch of mortar was used to mold nine 2-in. cubes for standard compressive strength tests in groups of three at 7, 28, and 91 days. Thus, the strength tests of the entire program involved over 5000 cubes from 576 batches, 48 for each sampling period.

A limited number of supplementary tests were made. Each monthly cement sample was tested for air-entraining capacity by ASTM Standard Method of Test for Air Content of Hydraulic Cement Mortar (C 185).[4] The cements representing the first two sampling periods were tested independently by two technicians to compare reproducibility and evaluate differences which might exist within the limits of the standard procedure. Finally, samples from 12 different locations within the same car were secured for two of the cements to provide an idea of variability within a single shipment. The 2 cements were the type I and type II from the same mill, mentioned earlier.

The investigation described here is to be contrasted with the earlier one in that: (1) 12 samples were secured for each of 14 cements instead of 26 for 5, (2) standard mortar tests only were made, whereas in the earlier investigation selected samples were also tested in a non-standard type of mortar and in two classes of concrete, and (3) in the earlier study, but not in this one, chemical analyses and fineness tests (air permeability), were made on selected samples. The concrete strength tests were omitted in the second program since the eariler one had established their close correlation with the strength of the standard mortar.

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[3] Method of Test for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. Cube Specimens) (C 109 - 58), 1958 Book of ASTM Standards, Part 4.

[4] Method of Test for Air Content of Hydraulic Cement Mortar (C 185 - 59), 1958 Book of ASTM Standards, Part 4.

TEST RESULTS

The test data are presented in Tables I to VI and Figs. 1 to 6. For the most part, only average results are given, but testing variability is indicated in representative cases by statistical measures. All strength results are averages for 9 cubes representing tests in triplicate from each of three batches made on different days. Flow and water content for the C

TABLE IA.—SUMMARY OF STRENGTH TESTS FOR CEMENT SOURCES 1 TO 7 (SERIES 168).

Each value average for 9 cubes; 3 from each of 3 batches mixed on different days, made and tested in accordance with ASTM Method C 109. The control sample was a blend of 5 brands, with 24 portions sampled and sealed at the same time. "Control A" designates portions mixed on the same days as sources 1 to 7; "Control B" designates like portions mixed on alternate mixing days with sources 8 to 14 (see Table IB). s_3 is sample-to-sample standard deviation in psi; v_3 is corresponding coefficient of variation in percentage. See Table IV for methods of calculating s and v.

Monthly Sample	Compressive Strength, psi							
	Source							
	1	2	3	4	5	6	7	Control Portion A
				7-DAY TESTS				
No. 1	2920	3402	3919	3299	3636	4218	3231	3626
No. 2	2774	3567	3437	3977	3584	4102	3308	3712
No. 3	3340	4034	3637	4047	3378	3761	3375	3857
No. 4	3152	3670	3834	4254	3372	3174	3426	3780
No. 5	3194	4203	3602	4040	3237	3491	2998	3877
No. 6	3178	4037	3536	3625	3278	3899	2199	3763
No. 7	2923	4056	3698	3593	3286	3908	3336	3910
No. 8	2828	4047	4096	3264	3212	4255	2926	3868
No. 9	3074	4219	4062	4372	3388	4422	3116	3951
No. 10	2981	3713	3994	4246	4026	4177	3069	3941
No. 11	3086	3844	3437	4050	3770	3875	3013	3837
No. 12	3694	3845	4079	4230	3048	4267	2514	4037
Avg	3095	3886	3778	3916	3434	3962	3042	3846
s_3, psi	250	257	251	378	273	359	364	113
v_3, per cent	8.1	6.6	6.6	9.6	7.9	9.1	12.0	2.9
				28-DAY TESTS				
No. 1	4167	4662	4842	4712	4701	5357	5558	4748
No. 2	3898	5034	4723	5526	4646	5440	5636	4928
No. 3	5127	5624	4937	5719	4560	4922	5818	5082
No. 4	4910	5381	5019	5765	4598	4701	6259	5027
No. 5	4681	5832	4771	5559	4337	4797	4839	5171
No. 6	4804	6025	4590	5514	4580	5156	4105	5229
No. 7	4455	6057	4954	5247	4595	5283	5637	5213
No. 8	4497	6105	5669	5103	4692	6122	5808	5393
No. 9	4727	6120	5328	5822	5064	5894	5676	5154
No. 10	4548	5434	5309	5945	4966	5751	5953	5279
No. 11	4383	5803	4796	5645	5130	5780	6156	5183
No. 12	5442	5611	5534	6185	4413	5876	5642	5359
Avg	4636	5641	5039	5562	4690	5423	5591	5147
s_3, psi	415	454	343	394	245	468	586	181
v_3, per cent	9.0	8.0	6.8	7.1	5.2	8.6	10.5	3.5

TABLE 1A.—*Continued.*

Monthly Sample	Compressive Strength, psi							
	Source							
	1	2	3	4	5	6	7	Control Portion A
	91-DAY TESTS							
No. 1	4792	5403	5305	5415	5271	5698	6578	5193
No. 2	4686	5558	4930	5825	5390	5701	6509	5251
No. 3	5761	6036	5248	6155	5234	5247	6285	5638
No. 4	5796	5932	5473	6224	5223	5117	7078	5417
No. 5	5546	6424	5179	6030	4938	5050	5743	5611
No. 6	5595	6558	5154	6343	5415	5485	5625	5824[a]
No. 7	5381	6524	5242	5850	5325	5644	6419	5594
No. 8	5315	6449	5685	5495	5383	6014	6270	5687
No. 9	5846	6468	5835	6237	6063	6313	6388	5519
No. 10	5510	6087	5904	6446	5803	6117	6914	5770
No. 11	5295	6474	5352	6407	5867	6333	7342	5780
No. 12	6083	6342	5874	6850	5254	6204	6735	5848
Avg	5467	6188	5432	6106	5431	5744	6491	5594
s_3, psi	412	389	321	410	320	457	499	215
v_3, per cent	7.5	6.3	5.9	6.7	5.9	8.0	7.7	3.8
	AVERAGE COEFFICIENT OF VARIATION, 3 TEST AGES							
v_3, per cent	8.2	7.0	6.4	7.8	6.3	8.6	10.1	3.4

[a] Average for 6 cubes only; one set of 3 accidentally destroyed.

TABLE IB.—SUMMARY OF STRENGTH TESTS FOR CEMENT SOURCES 8 TO 14 (SERIES 168).

Each value average for 9 cubes; 3 from each of 3 batches mixed on different days, made and tested in accordance with ASTM Method C 109. The control sample was a blend of 5 brands, with 24 portions sampled and sealed at the same time. "Control B" designates portions mixed on the same days as sources 8 to 14; "Control A" designates like portions mixed on alternate mixing days with sources 1 to 7 (see Table IA). s_3 is sample-to-sample standard deviation in psi; v_3 is corresponding coefficient of variation in percentage. See Table IV for methods of calculating s and v.

Monthly Sample	Compressive Strength, psi							
	Source							
	8	9	10	11	12	13	14	Control Portion B
	7-DAY TESTS							
No. 1	2827	3057	3668	3519	2932	3667	2951	3482
No. 2	2861	3456	4169	4428	3314	3496	3246	3693
No. 3	2919	3377	3914	3774	3222	3750	3372	3729
No. 4	...	3234	3925	3965	3051	3844	2857	3777
No. 5	2885	3394	3825	4032	2695	3488	2995	3884
No. 6	3103	3083	3384	4088	2801	3833	3373	3800
No. 7	2876	3022	4592	4156	2802	3366	2449	3828
No. 8	2934	3107	4153	4396	2418	3884	2562	3850
No. 9	2654	3425	3769	4057	3153	3898	2681	3952
No. 10	3025	3501	3731	4488	2699	4348	2387	3997
No. 11	2941	3533	4031	4643	2473	3934	2915	3866
No. 12	3211	4066	4039	3994	3093	3863	2713	3916
Avg	2931	3354	3933	4128	2888	3781	2875	3814
s_3, psi	147	290	303	318	294	258	336	137
v_3, per cent	5.0	8.6	7.7	7.7	10.2	6.8	11.7	3.6

TABLE 1B.—*Continued.*

Monthly Sample	Compressive Strength, psi							
	Source							
	8	9	10	11	12	13	14	Control Portion B
28-DAY TESTS								
No. 1	4773	4347	5651	5692	4051	5153	4233	4762
No. 2	4837	5235	5808	6079	4607	4983	4709	4871
No. 3	4983	4863	5727	5858	4456	5392	4624	4851
No. 4	...	4736	5973	6130	4500	5432	4308	5104
No. 5	4943	5163	5805	5699	4185	5182	4310	5071
No. 6	4901	4374	5163	5862	5157	5500	4893	5160
No. 7	4797	4459	6357	6646	4017	4715	3738	5105
No. 8	4926	4639	6011	6813	3716	5686	4114	5467
No. 9	4502	4866	5731	6431	4634	5668	4389	5185
No. 10	5227	5354	5885	6492	4158	5907	4038	5187
No. 11	5260	5318	6081	6901	3968	5681	4607	5188
No. 12	5617	6048	6168	6517	4685	5712	4364	5220
Avg	4979	4950	5863	6260	4344	5418	4361	5098
s_3, psi	297	498	301	429	400	350	317	192
v_3, per cent	6.0	10.1	5.1	6.8	9.2	6.4	7.3	3.8
91-DAY TESTS								
No. 1	5626	5392	6803	6665	5202	5695	5134	5319
No. 2	5552	5902	6621	6470	5867	5468	5396	5299
No. 3	5957	5774	6705	6814	5581	5788	5502	5380
No. 4	...	5789	6940	7016	5658	6015	5355	5664
No. 5	5987	6290	6873	6539	5979	5941	5386	5651
No. 6	6021	5776	6419	6796	6664	6090	5756	5736
No. 7	5934	5628	7315	7369	5539	5212	4836	5655
No. 8	5856	5608	7047	7790	5097	5868	5342	5693
No. 9	5655	6340	6835	7385	6167	6193	5846	5556
No. 10	6319	6526	7073	7396	5941	6521	5634	5837
No. 11	6380	6394	7376	7795	5551	6207	5876	5881
No. 12	6193	6804	7697	7512	6168	6637	5991	5584
Avg	5953	6018	6975	7129	5784	5970	5504	5604
s_3, psi	273	436	354	471	439	406	334	189
v_3, per cent	4.6	7.2	5.1	6.6	7.6	6.8	6.1	3.4
AVERAGE COEFFICIENT OF VARIATION, 3 TEST AGES								
v_3, per cent	5.2	8.6	6.0	7.0	9.0	6.7	8.4	3.6

109 mortars are averages for the three batches. Data for the air content tests represent single batches.

Within-Source Strength Variations:

Table I gives the compressive strengths. Portions A and B of the table separate the two groups of 7 cements which, as noted above, were tested on different days. It is significant that the control cement gave almost identical strengths in both groups, indicating the across-the-board comparability of results in spite of the necessary time separation.

All cements from outside sources were more variable in mortar strength than the control cement. Average coefficients of variation for the three test ages ranged from 5.2 per cent for source 8 to 10.1 per cent for source 7, compared with an average of 3.5 per cent for the two sets of control data.

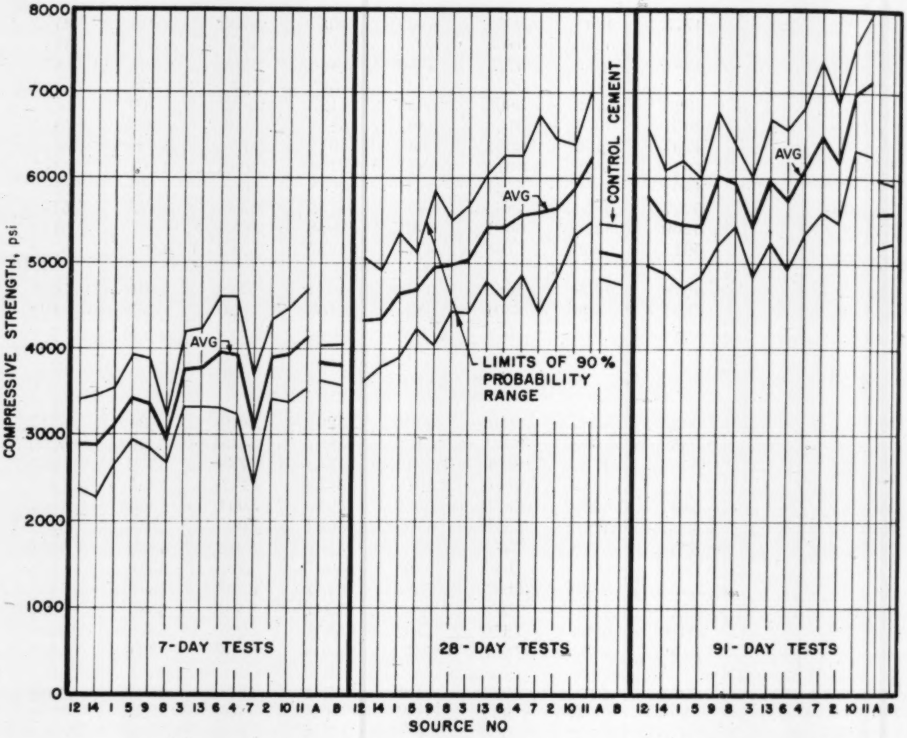

FIG. 1.—Source-to-Source and Within-Source Variations in C 109 Mortar Strength of Cements (Series 168).

Accepted statistical procedures indicate that the coefficients of variation for the outside cements are significantly higher than the control. Even for the most uniform of the cements, the chances are less than 1 in 40 that this is not so. It is of interest to point out that the within-source uniformity in the current investigation is only slightly better than in the earlier one.

Figure 1 shows strength variations between and within sources. The heavy line gives averages for each source in ascending order of 28-day strength. The lighter lines represent the range within which the strength would be expected to fall 90 per cent of the time. (For a population of 12 samples, that range is 1.80*s* where *s* is the standard deviation.) It follows, of course, that one sample in 20 would be expected to fall below the lower limit of Fig. 1.

As already pointed out, earlier work has demonstrated that differences in mortar strength accurately reflect differences in concrete strength. For the two most uniform cements, Nos. 5 and 10, with coefficients of variation of about

5 per cent at 28 days, the strength range expected to encompass 90 per cent of all samples would be ± 9 per cent of the average. Thus, about 1 time in 20 the strength would be more than 9 per cent below the average. For the control cement the prediction would be that 1 in 20 would be about 6.5 per cent or more below the average and only about 1 in 100 more than 9 per cent below. Cements Nos. 7 and 9, with coefficients of variation of over 10 per cent at 28 days, would be expected to fall more than 18 per cent below their own averages once in 20 times.

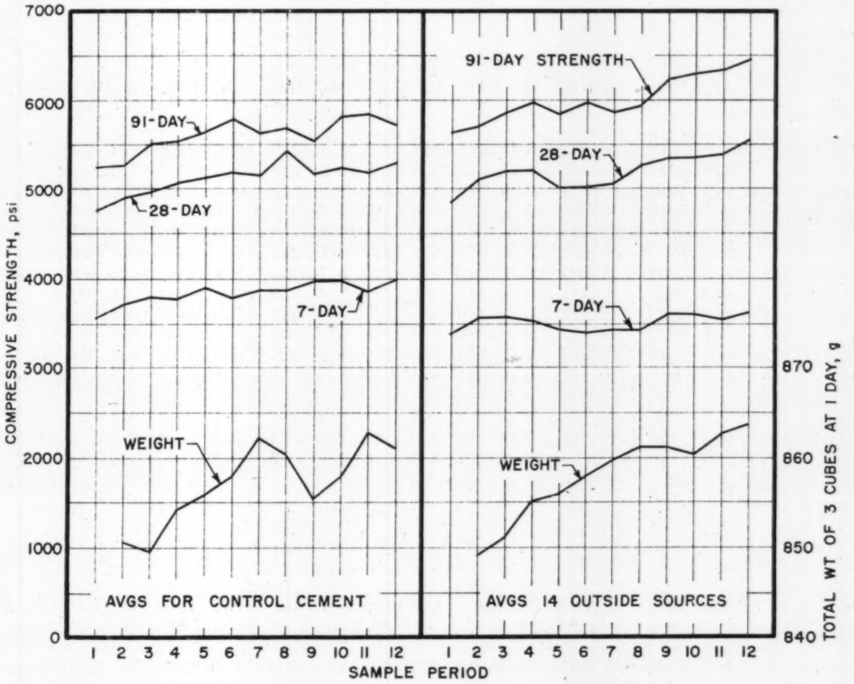

FIG. 2.—Weight and Strength Trends for Mortar Cubes (Series 168).

The uniformity of cement depends on many factors, some of which are more readily controlled than others. Both operating procedures and uniformity of raw materials enter into the picture. It does not follow, therefore, that the mills showing poorest uniformity can, by economically feasible operating changes, be made to match the performance of those with best uniformity. Nevertheless, room for improvement exists. It should be of interest that, using the low average coefficient of 5.2 for source 8 as a basis for comparison, statistical analyses indicate a probability of better than 95 in 100 that sources 1, 4, 6, 7, 9, 12, and 14 were actually more variable than source 8.

Time Effects:

Since these tests were performed over a period of a year, the comparability of results representing the different sampling periods is of major importance. This, of course, was a principal reason for including the control cement, which should be expected to reveal variations in testing independently of differences in cement. Data related to this question are shown in Fig. 2, where strengths are arranged in chronological order of testing for the control cement and for the averages of all other cements. Also shown

TABLE II.—AIR CONTENT TESTS OF MORTAR (SERIES 168).

Tests made in accordance with Standard Method of Test for Air Content of Hydraulic Cement Mortar, ASTM Designation C 185 – 59.[4] Flow maintained within prescribed range of 80 to 95 per cent except as noted. Tabulated mixing water is that required in batch containing 350 g cement and 1400 g Standard (No. 20–30) sand.

Monthly Sample	Cement Source No.															
	1		2		3		4		5		6		7		Control	
	Air, %	H_2O, ml	Air, %	H_2O, ml	Air, %	H_2O, ml	Air, %	H_2O, ml	Air, %	H_2O, ml	Air, %	H_2O, ml	Air, %	H_2O, ml	Air, %	H_2O, ml
No. 1	11.6	220	9.4	235	8.0	235	11.8	225	11.1	230	9.4	240	11.4	220	...	...
No. 2	13.0	218	9.5	232	8.7	245	10.0	232	10.6	232	9.5	240	11.9	225	...	...
No. 3	9.4	230	8.6	235	10.6	242	8.9	235	8.6	235	9.5	242	10.0	235	...	...
No. 4	9.1	232	9.1	235	10.1	235	8.6	240	9.5	235	8.7	240	9.8	232	...	...
No. 5	8.8	230	9.1	235	10.1	242	11.6	235	10.6	235	9.3	240	10.2	235	...	...
No. 6	9.9	242	8.8	235	8.8	242	8.2	242	8.8	242	8.6	242	9.6	242	10.2	235
No. 7	11.3[a]	242[a]	8.7[b]	235[b]	8.9	242	8.6	242	9.7	235	8.7	245	10.8	230	9.4	235
No. 8	9.3	239	7.8	242	8.5	242	9.8	246	9.4	239	9.9	246	9.6	239	9.1	239
No. 9	8.0	242	8.1	242	10.5	246	7.5	246	7.6	242	8.9[a]	249[a]	10.1	239	9.1	242
No. 10	8.5	242	8.1	242	9.9	246	8.6	244	9.2	242	9.1	248	8.1	248	8.3	242
No. 11	8.6	240	7.8	243	9.2	244	8.2	242	8.5	244	8.5	244	8.5	243	9.2	240
No. 12	8.0	242	7.8	242	10.2	242	8.5	240	8.9	240	8.5	242	8.2	240	8.8	240
Avg	9.6	235	8.6	238	9.5	242	9.2	239	9.4	238	9.1	243	9.8	236	9.2	239
Range	5.0	24	1.7	11	2.6	11	4.3	21	3.5	14	1.4	9	3.8	28	1.9	7
s_3	1.6	9	0.6	4	0.9	4	1.3	6	1.0	5	0.5	3	1.2	8	0.7	3

Monthly Sample	Cement Source No.															
	8		9		10		11		12		13		14		Average[c]	
	Air, %	H_2O, ml	Air, %	H_2O, ml	Air, %	H_2O, ml	Air, %	H_2O, ml	Air, %	H_2O, ml	Air, %	H_2O, ml	Air, %	H_2O, ml	Air, %	H_2O, ml
No. 1	8.2	235	8.0	225	6.6	235	7.7	235	8.5	235	7.9	245	6.7	235	9.0	232
No. 2	9.0	235	9.4	225	8.4	232	9.2	240	11.0	220	10.1	235	8.6	232	9.9	232
No. 3	8.9	240	9.7	230	8.4	245	9.2	242	9.7	230	9.0	245	8.7	240	9.2	238
No. 4	...	...	9.4	230	8.2	235	8.7	232	9.3	228	7.9	242	8.4	240	9.0	235
No. 5	11.0	235	8.7	235	9.4	235	8.3	235	9.4	230	8.0	245	8.6	242	9.5	236
No. 6	8.6	242	8.9	232	8.6	235	7.8	242	11.9	227	8.6	245	8.9	235	9.1	239
No. 7	8.9	242	8.6	232	7.8	235	9.2	242	9.3	230	9.6	235	8.6	235	9.2	237
No. 8	8.6	242	8.5	235	7.9	242	9.2	239	8.3	249	9.1	246	8.1	242	8.9	242
No. 9	8.0[a]	249[a]	8.9	246	6.7	250	7.3	250	10.1[a]	246[a]	7.8	248	7.6	248	8.4	246
No. 10	8.4	242	7.8	242	7.3	242	8.0	248	9.8	246	9.8	246	8.4	246	8.7	244
No. 11	8.5	239	8.3	239	8.1	240	8.0	240	8.9	248	8.6	244	7.9	243	8.5	242
No. 12	...	...	7.6	240	8.3	238	7.8	240	8.3	238	7.7	244	7.6	242	8.3	241
Avg	8.8	240	8.7	234	8.0	237	8.4	240	9.5	236	8.7	243	8.3	240	9.0	239
Range	3.0	14	2.1	21	2.8	18	1.9	18	3.6	29	2.4	13	2.2	16	1.6	14
s_3	0.8	4	0.7	7	0.8	6	0.7	5	1.1	10	0.8	4	0.7	5	0.5	5

[a] Flow 95 to 100 per cent.

[b] Flow 79 per cent.

[c] Average of 14 sources.

are the average one-day weights of cubes measured in groups of three from the same gang mold when the specimens were stripped.

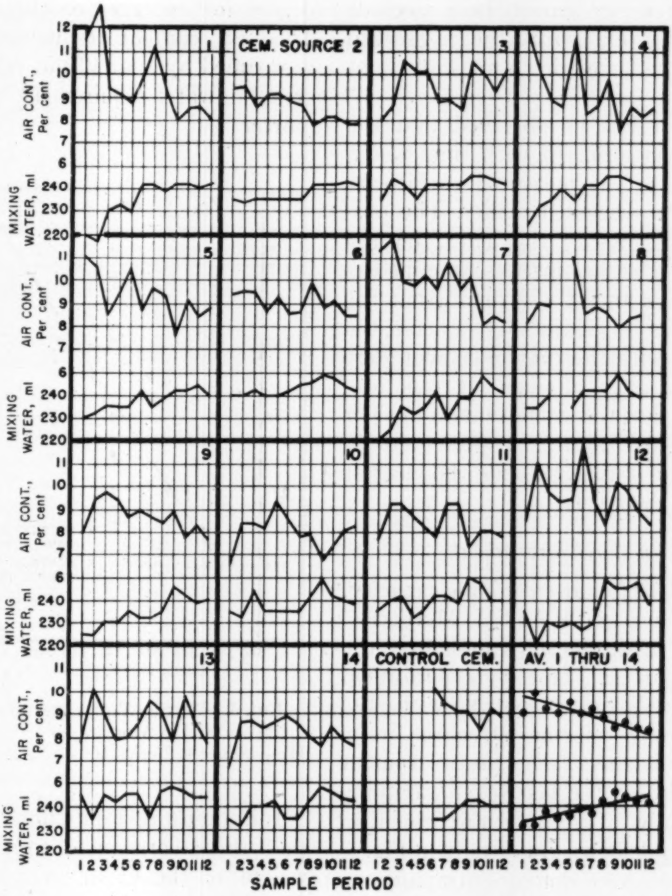

FIG. 3.—Variations in Air Content and Mixing Water of C 185 Mortar for Successive Samples of Cement from Different Sources (Series 168).

Both strength and cube weight increased progressively with the passage of time. The trends were of similar magnitude for the control cement and the cements from outside sources, indicating a progressive alteration in some aspect of testing. The direction of the trend in both strength and weight is consistent with the hypothesis that specimens became denser (contained less air) as the work progressed. Analyses of standard air content measurements on the cements, given in Table II and Fig. 3, indicate that the increase in density is probably attributable to a progressive decrease in mortar air content, possibly caused by a change in the standard sand.

Whatever the cause, if it is accepted that the changes in weight reflected changes in density (rather than dimensions of cubes), then the range corresponds to a difference in void content of about 1½ per cent during the period of testing. Data of several investigators have shown that strength is reduced 5 per cent or more for each per cent of added air when the water-cement ratio is unchanged. Thus, the 1½ per cent variation in density shown here accords reasonably well with the observed strength trend, covering a range of about 10 per cent.

It cannot but be observed that the variations in strength with time would contribute to the measured coefficients of variation discussed earlier. The possible significance of this effect on the statistical relationships was examined in several ways and it was found that the comparisons of variability among the sources or with the control cement were not significantly affected. It was felt, therefore, that the strength data in their unembellished form, as presented in Table I and Fig. 1, provide the clearest picture of the results.

Variations in Air Entrainment:

Single tests for air-entraining capacity, in accordance with ASTM Method C 185,[4] were made on each sample from the outside sources. These were performed approximately concurrently with the corresponding mortar strength tests. Air content tests of the control cement were made for the last seven sampling periods only. The data are given in Table II and shown graphically in Fig. 3. All cements were non-air-entraining according to ASTM standards. Only in the case of a single sample of cement No. 1 was the 12 per cent maximum of ASTM Specification C 150[5] barely exceeded.

There was variation in the air content from sample to sample for a given cement source. This may have been partly due to testing variation, since each sample was represented by only a single test. However, there is a clearly evident trend toward decreasing air content for successive sampling periods, discernible in Fig. 3, to some degree for several individual sources and to a very impressive degree for averages of the 14 sources. The air content reduction was accompanied by a small but consistent increase in water requirement of the C 185 mortar.

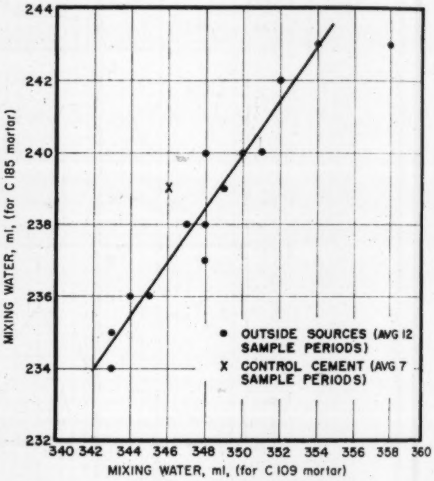

FIG. 4.—Mixing-Water Relationship Between C 109 and C 185 Mortars for Cement from Different Sources (Series 168).

It is almost certainly not a coincidence that these correspond to the trend in weight of the C 109 mortar discussed above. In fact, the range in air content, shown by the curve for averages in Fig. 3 to be about 1½ per cent, checks almost exactly the air content differences mentioned earlier as accounting for the change in cube weights. The air content test data, therefore, lend support to the conjecture that the strength and weight trends for C 109 cubes were caused by changes in air content of the mortar.

The cause of the air content trend is

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[5] Specification for Portland Cement (C 150 – 60), 1960 Supplement to the Book of ASTM Standards, Part 4.

not known. A study of 150 sieve analyses of the sand, representing tests in duplicate on each of 75 bags used, denied that grading was responsible. Average fineness moduli of the graded sand ranged only from 1.73 to 1.76 for the 12 sampling periods. Other possible sources of progressive air content differences suggest themselves, but these are not easy to document or support.

TABLE III.—MIXING WATER REQUIREMENTS OF MORTAR (SERIES 168).

Tabulated values are mixing water in ml for standard 9-cube batch (with 740 g cement and 2035 g graded standard sand) and flow of mortar in per cent (25 drops of table in 15 sec). Each value average for 3 batches mixed on different days.

Monthly Sample	Cement Source No.															
	1		2		3		4		5		6		7		Control Portion A	
	H_2O, ml	Flow, %	H_2O, ml	Flow, %	H_2O, ml	Flow, %	H_2O, ml	Flow, %	H_2O, ml	Flow, %	H_2O, ml	Flow, %	H_2O, ml	Flow, %	H_2O, ml	Flow, %
No. 1	335	106	345	108	350	108	340	104	345	106	355	107	335	105	345	108
No. 2	330	105	343	108	358	106	343	106	343	110	353	109	338	109	345	106
No. 3	333	105	345	108	357	109	343	103	345	106	355	110	343	102	348	111
No. 4	340	110	345	107	345	109	347	106	345	106	350	108	337	105	343	106
No. 5	335	106	345	107	345	107	345	111	345	106	350	107	347	109	345	108
No. 6	350	103	347	111	348	103	348	104	350	110	355	106	350	110	347	108
No. 7	352	110	348	107	352	105	350	107	348	110	353	108	338	104	345	105
No. 8	347	108	348	107	350	107	353	106	345	105	355	106	343	102	345	105
No. 9	353	107	353	110	357	105	357	107	350	109	360	110	345	108	348	106
No. 10	350	109	348	112	350	109	362	105	355	113	360	107	358	107	350	109
No. 11	345	106	355	112	357	110	350	110	347	109	357	109	355	108	345	108
No. 12	350	106	350	109	350	108	348	106	343	108	350	107	348	107	345	106
Avg	343	106	348	108	352	107	349	106	347	108	354	108	345	106	346	107
Range	23	7	12	5	13	7	22	8	12	8	10	4	23	8	7	6
Per cent range	6.7	6.6	3.5	4.6	3.7	6.5	6.2	7.5	3.5	7.4	2.8	3.7	6.7	7.5	2.0	5.6

	Cement Source No.															
	8		9		10		11		12		13		14		Control Portion B	
	H_2O, ml	Flow, %	H_2O, ml	Flow, %	H_2O, ml	Flow, %	H_2O, ml	Flow, %	H_2O, ml	Flow, %	H_2O, ml	Flow, %	H_2O, ml	Flow, %	H_2O, ml	Flow, %
No. 1	345	109	340	107	347	108	350	105	350	104	360	108	347	108	342	106
No. 2	350	110	340	106	345	106	355	108	335	112	353	107	345	109	345	106
No. 3	350	110	343	108	347	107	355	108	333	104	365	106	348	105	347	106
No. 4	...	...	337	108	345	107	342	110	330	106	355	107	348	106	345	109
No. 5	340	108	345	107	345	108	345	105	335	109	358	104	353	104	345	106
No. 6	348	106	343	111	345	104	347	106	332	110	358	106	345	105	347	108
No. 7	348	107	340	106	345	103	352	106	335	106	347	107	345	106	345	105
No. 8	350	107	340	106	350	105	343	108	360	107	353	104	352	108	345	105
No. 9	360	107	353	105	362	104	360	108	362	109	362	105	365	112	348	104
No. 10	350	109	348	105	362	107	357	109	353	104	365	104	362	108	352	111
No. 11	343	109	342	108	345	109	345	107	370	108	358	106	355	108	345	105
No. 12	345	107	345	109	342	107	345	110	338	111	357	110	350	109	342	105
Avg	348	108	343	107	348	106	350	107	344	107	358	106	351	107	346	106
Range	20	4	17	6	20	6	18	5	40	8	18	6	20	8	10	7
Per cent range	5.8	3.7	5.0	5.6	5.8	5.7	5.1	4.7	11.6	7.5	5.0	5.7	5.7	7.5	2.9	6.6

Laboratories on occasion have encountered air-entraining properties in the standard sand. It is conceivable that the substances causing the air entrainment might lose their potency with time. Other possibilities, such as changes in performance of equipment or personnel, can be hypothesized as being responsible but, so far, none has seemed sufficiently tenable to merit discussion here.

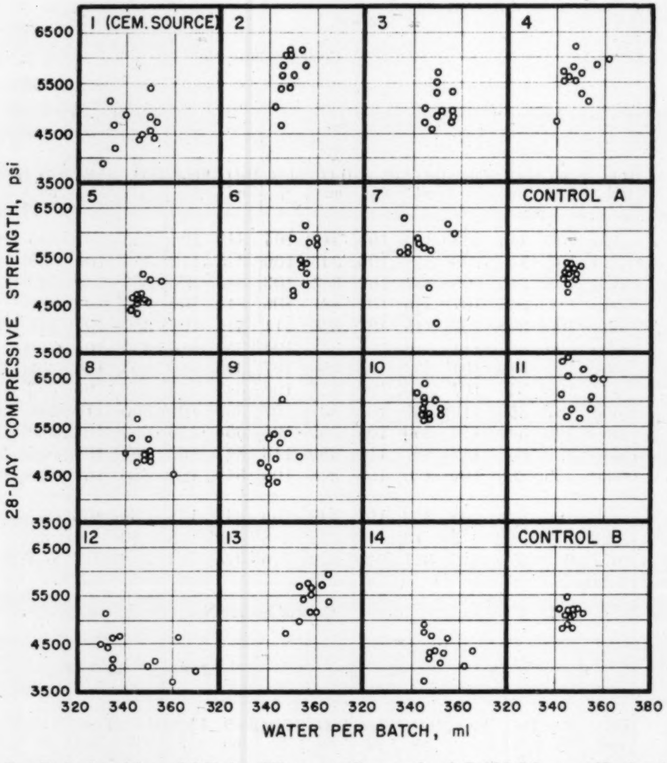

Fig. 5.—Relationships of Mixing Water to Strength of C 109 Mortar (Series 168).

Independently of the long-range trend of air contents with time, it should be observed that the individual fluctuations in air produced by different samples of a given cement appear to be real. Multiple tests on the same shipment of cement, discussed later, showed the test method for air content to be highly reproducible, suggesting that the within-source variations shown in Fig. 3 cannot be attributed to testing error. As a further check on the reliability of the measurements, air content tests were repeated at the close of the program on pairs of samples representing extremes of air in the original tests for four of the cements. In all cases, the pairs produced air content differences in the right direction and consistent in magnitude with the original tests.

An item of by-product data from the air content tests is shown in Fig. 4. Although differences in mixing water among the different cement sources were small, the requirements for C 109 and C 185 mortar correlated very closely.

Variations in Water Demand:

Table III shows average flows and mixing water contents for the three batches of mortar made with each cement sample. All flow values are within the range of 100 to 115 specified in Method C 109, and averages for individual sources vary only from 106 to 108. No relationship was found between mixing water and the small flow variations within the permissive range for individual cement sources. It may be inferred, therefore, that mixing water differences for a given source represent the combined effects of testing error and

TABLE IV.—TESTS OF WITHIN-CAR UNIFORMITY OF CEMENT STRENGTH (SERIES 168).

Tests by ASTM Method C 109 of samples from 12 different locations in cars of cement from 2 sources. Each location represented by average of 3 batches mixed on different days, with each strength the average of nine 2-inch cubes—3 from each batch.

Sample Location in Car	Results of C 109 Mortar Tests									
	Cement Source No. 6					Cement Source No. 7				
	Water, ml	Flow, per cent	Compressive Strength, psi (or per cent)			Water, ml	Flow, per cent	Compressive Strength, psi (or per cent)		
			7-day	28-day	91-day			7-day	28-day	91-day
a	350	106	4407	5795	5919	350	105	3820	6331	7102
b	350	107	4372	5781	5951	350	106	3812	6367	7146
c	350	106	4373	5844	6003	350	108	3610	6373	7144
d	350	107	4539	5877	6048	350	107	3809	6188	7156
e	350	107	4448	5696	6082	350	108	3678	6382	7179
f	350	106	4433	5812	5969	350	108	3657	6244	7076
g	350	106	4504	5783	6019	350	105	3672	6361	7060
h	350	107	4347	5608	5922	350	104	3799	6463	7217
k	350	106	4346	5741	5949	352	107	3649	6407	7171
l	350	106	4432	5651	5818	352	106	3673	6321	7109
m	350	107	4352	5719	5961	353	108	3620	6435	7303
n	350	106	4492	5736	5799	353	108	3685	6287	7150
Avg	350	106	4420	6754	5956	351	107	3707	6347	7151
s_3, psi[a]			63	78	80			80	78	65
v_3, per cent[a]			1.4	1.4	1.3			2.1	1.2	0.9
$C_{a\text{-}f}$[b]	347	108	3911	5265	5656	348	109	3846	5247	5733
$C_{g\text{-}n}$[b]	348	109	3836	5138	5654	350	109	3848	5218	5897
Avg	348	108	3874	5202	5655	349	109	3847	5232	5815
s_1, psi[a]			87	165	116			86	129	121
s_2, psi[a]			104	124	152			79	113	142
v_1, per cent[a]			2.0	2.9	1.9			2.3	2.0	1.7
v_2, per cent[a]			2.4	2.2	2.5			2.1	1.8	2.0

[a] s_1 = within batch standard deviation = $0.5907\overline{R}$ where $\overline{R}$ is average range for 42 sets of 3 cubes representing a given cement and its control batches at a given age.

s_2 = batch-to-batch standard deviation = $0.5907\overline{R}$ where $\overline{R}$ is average range in strength for the 14 sets of 3 batches representing given samples of the same cement and its control batches at a given age.

s_3 = sample-to-sample standard deviation = $\sqrt{\frac{\Sigma(\bar{x} - x)^2}{n - 1}}$ where $\bar{x}$ is average strength at a given age, x is the strength of a particular sample and n is the number of samples, 12.

v_1, v_2 and v_3 are corresponding coefficients of variation expressed as percentages of average strengths.

[b] Batches with control cement, mixed concurrently with samples a through f or samples g through n of the given source of cement.

variation in the water demand of the cement.

The range in mixing water was greater for all outside sources than the 10-ml difference exhibited by the control. Ranges exceeding twice that amount, 20 ml, are indicated for cements Nos. 1, 4, 7, and 12. The latter showed by far the greatest range, 40 ml, amounting to nearly 12 per cent of the average water content.

As shown in Fig. 5, there was generally no indication that strength variations of the mortar were related to the differences in water demand. Only in the case of source 12, mentioned above as revealing greatest variation in mixing water, was there a tendency for higher water requirement to be associated with lower strength.

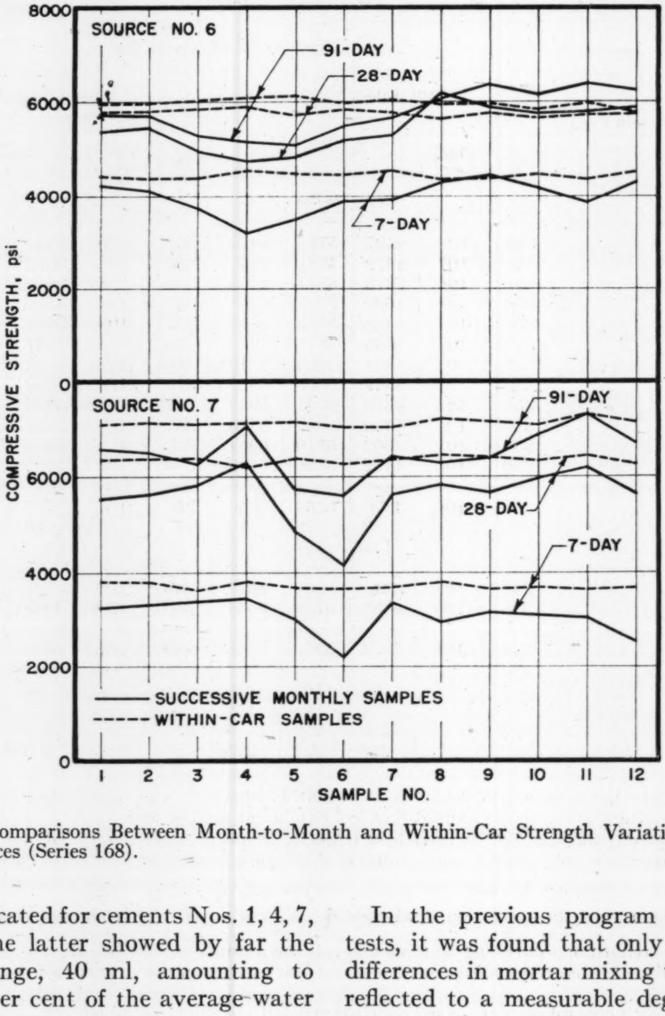

FIG. 6.—Comparisons Between Month-to-Month and Within-Car Strength Variations for Two Cement Sources (Series 168).

In the previous program of cement tests, it was found that only very large differences in mortar mixing water were reflected to a measurable degree in the water requirement of concrete. With the possible exception of cement source 12, which exhibited the greatest variability, it is doubtful that differences in water demand found in the present tests would significantly affect the required mixing water or slump of concrete.

Tests of Within-Shipment Uniformity:

An additional basis for comparison with the tests on monthly samples was provided by securing several samples from single shipments. Samples were taken from 12 locations within single cars of two cements, Nos. 6 and 7. These were, respectively, a type I and a type II from the same mill.

TABLE V.—AIR CONTENT VARIATIONS FOR CEMENT SAMPLES FROM SAME CAR (SERIES 168).

Tests made in accordance with ASTM Method C 185 – 59.

Quantities for all batches: cement 350 g; Standard Sand (No. 20–30) 1400 g; water 242 ml.

Data represent single batches: source 6 plus control on one day; source 7 plus control on second day.

Sample Location in Car	Cement Source No. 6		Cement Source No. 7	
	Air Content, per cent	Flow, per cent	Air Content, per cent	Flow, per cent
a	8.0	88	9.2	91
b	8.7	91	9.5	90
c	8.9	90	9.1	90
d	8.6	87	9.2	91
e	8.9	90	9.8	91
f	8.5	85	9.8	92
g	8.8	92	9.7	90
h	8.7	87	9.9	95
k	8.2	88	9.7	90
l	8.7	86	10.0	89
m	8.4	88	9.7	87
n	8.8	88	10.0	87
Avg	8.6	88	9.6	90
Control	9.8	95	9.0	93

The samples were tested using procedures similar to those of the main program except that all mortar was mixed within a short period of time. The 24 samples, 12 from each car, were mixed in groups of six, together with the control cement, on each of four days. Three such rounds were made. The average data are given in Table IV. In Fig. 6, strengths for 12 samples from the same shipment are compared graphically with 12 samples taken at one-month intervals from the same sources.

The data indicate that there was no significantly measurable variation in the samples from the same shipment. The sample-to-sample coefficients of variation, averaging 1.4 per cent for both sources, are almost precisely what would be expected if the tests had all been made on the same sample. Thus, within the precision of the meticulously controlled methods of this investigation, the cement in each of the two cars sampled was uniform. The 1500 to 2000 psi spread in 28-day strengths of samples from different shipments shown in Fig. 6 contrasts sharply with the 270 to 275 psi within-car range.

Air content variations within a particular car of cement were very small as shown in Table V. The ranges and standard deviations indicate good precision of the test method, at least for determinations made within a short period. It seems probable, therefore, that the reliability of the air content measurements on the monthly samples, discussed earlier, would not have been appreciably improved by making replicate determinations.

Source-to-Source Variations:

The average 28-day strengths of the 12 samples for each of the 14 cements ranged from 4344 psi for source 12 to 6260 psi for source 11, a spread of about 36 per cent of the mean. This is only slightly less than the difference shown for 28-day tests of the five sources in the earlier investigation in which the low value was 3674 psi and the high 5434 psi, a spread of 39 per cent.

For the current tests at 7 days, the

TABLE VI.—COMPARISON OF MORTAR TESTS BY TWO OPERATORS (SERIES 168).

Values are averages for 3 batches mixed on different alternate days by each operator, with 9 cubes molded from each batch for tests in triplicate at 3 ages. s_1 and s_2 are within-batch and batch-to-batch standard deviation and v_1 and v_2 are the corresponding coefficients of variation in percentage (see Table IV for bases of calculation).

Cement Source	Tests by Operator No. 1						Tests by Operator No. 2					
	Water, ml	Flow, per cent	Compressive Strength, psi			Average Weight 3 Cubes, g[a]	Water, ml	Flow, per cent	Compressive Strength, psi			Average Weight 3 Cubes, g[a]
			7 days	28 days	91 days				7 days	28 days	91 days	
Sample Period No. 1												
No. 1	335	106	2920	4139	4802		335	107	2860	3934	4940	
No. 2	345	108	3391	4662	5403		350	111	3359	4550	5463	
No. 3	350	108	3919	4842	5305		350	110	3869	4751	5377	
No. 4	340	104	3299	4712	5412		340	108	3427	4840	5619	
No. 5	345	106	3636	4701	5271		347	108	3711	4560	5359	
No. 6	355	107	4218	5357	5698		353	108	4162	5139	5729	
No. 7	335	105	3231	5558	6578		335	106	3097	5547	6657	
No. 8	345	109	2827	4773	5626		350	110	2869	4766	5698	
No. 9	340	107	3057	4347	5392		340	107	3100	4618	5618	
No. 10	347	108	3668	5651	6803		350	106	3585	5492	6721	
No. 11	350	105	3519	5692	6665		350	104	3432	5595	6620	
No. 12	350	104	2932	4051	5202		350	105	2917	4080	5250	
No. 13	360	108	3667	5153	5695		360	108	3620	4997	5618	
No. 14	347	108	2985	4233	5134		350	108	2917	4262	5156	
C_{1-7}[b]	345	108	3626	4748	5193		343	107	3544	4554	5321	
C_{8-14}[b]	342	106	3549	4762	5319		343	106	3550	4745	5344	
Avg	346	107	3403	4836	5594		347	107	3376	4777	5656	
s_1	...	...	89	119	136		...	...	112	153	134	
s_2	...	...	156	138	167		...	...	87	137	117	
v_1	...	...	2.6	2.5	2.4		...	...	3.3	3.2	2.4	
v_2	...	...	4.6	2.9	3.0		...	...	2.6	2.9	2.1	
Sample Period No. 2												
No. 1	330	105	2774	3898	4686	831	332	110	2676	3983	4838	823
No. 2	343	108	3567	5034	5558	856	343	109	3525	5187	5879	852
No. 3	358	106	3437	4723	4930	836	353	104	3445	4702	5054	830
No. 4	343	106	3950	5526	5870	852	340	108	3913	5680	6175	849
No. 5	343	110	3593	4691	5390	847	345	111	3523	4648	5271	839
No. 6	353	109	4097	5440	5701	851	355	112	4170	5514	5729	846
No. 7	338	109	3308	5670	6509	852	335	109	3393	5934	6779	846
No. 8	350	110	2861	4837	5552	850	350	110	2823	4979	5479	846
No. 9	340	106	3484	5235	5902	854	340	105	3407	5186	6215	852
No. 10	345	106	4169	5808	6621	857	350	112	3955	5918	6580	853
No. 11	355	108	4428	6079	6470	854	353	106	4419	6143	6652	850
No. 12	335	112	3314	4607	5867	847	335	110	3226	4876	5923	838
No. 13	353	107	3496	4983	5468	849	360	111	3361	4909	5456	840
No. 14	345	109	3246	4709	5396	852	350	112	3172	4769	5619	842
C_{1-7}[b]	345	106	3712	4928	5251	852	345	107	3652	4992	5234	846
C_{8-14}[b]	345	106	3693	4871	5299	849	345	108	3703	5063	5503	844
Avg	345	108	3571	5065	5651	849	346	109	3523	5155	5774	843
s_1	...	...	77	116	148		...	...	103	120	146	
s_2	...	...	84	181	147		...	...	146	233	205	
v_1	...	...	2.2	2.3	2.6		...	...	2.9	2.3	2.5	
v_2	...	...	2.4	3.6	2.6		...	...	4.1	4.5	3.5	

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[a] Cubes weighed 3 at a time (from same gang mold); each value average of the 9 weighings representing a given source.

[b] Control batches mixed same day as sources 1 through 7 and sources 8 through 14, respectively.

percentage spread was about 36 per cent, the same as for 28 days, but at 91 days the variation was reduced somewhat, to about 27 per cent. It appears that there was some tendency for age to ameliorate the early strength differences among cement sources.

The presence of the type II cements in this study had no effect on the over-all range among sources. They were Nos. 7, 8, 9, and 14, none of which represented strength extremes. They did, however, alter the relative arrangement of strength levels at the different ages, as evidenced by the markedly low values at 7 days for Nos. 7 and 8 and the perhaps higher than expected levels for Nos. 7, 8, and 9 in relation to the others at 91 days.

Differences in average water requirement among the cement sources were not large, encompassing an over-all range of only 343 to 358 ml for the standard 9-cube batches. There was no discernible relationship between water demand and strength level.

COMPARISONS BETWEEN OPERATORS

When the testing program was being organized, it was decided to assign the mixing and testing of C 109 mortars to a different operator from the one who had performed those duties in the previous research. Therefore, for the first two sample periods, the tests were run in parallel by both men—operator No. 1 being the one who performed the C 109 tests for this report, and operator No. 2 for the previous program. The results are compared in Table VI.

On the whole, the two men provided virtually identical results. Mixing water contents were essentially the same even though there was no consultation or comparison between operators in selecting the amounts. Strengths for individual sources checked very closely; over-all averages at any age for either sampling period lie within a range of 123 psi or less. Even these very small differences in strength were not consistently in the same direction. For the six pairs of comparable average strengths (three ages, two sample periods), each operator was higher than the other three times. The average difference for the 96 pairs of strength comparisons amount to less than $1\frac{1}{2}$ psi.

In spite of slight differences one way or the other for individual groups of comparable data, there appeared to be no over-all difference between the two operators in uniformity of strength tests. Average within-batch coefficients of variation were 2.4 and 2.8 per cent for operators 1 and 2, respectively. Corresponding batch-to-batch values were 3.2 and 3.3 per cent.

Weighing of cubes, which was not initiated until sample period 2, showed slightly but consistently higher levels for operator No. 1. The difference averaged 0.7 per cent. It is not known whether this represented a difference in the compactive effort of molding specimens or, perhaps, a consistent difference in striking them off.

The comparative data provided assurance that the new operator could perform the tests skillfully and reproducibly, with results comparable in significance to those from the earlier project. They are also of interest in suggesting that Method C 109 is sufficiently well delineated to assure that different skilled operators will produce the same results when working under the same laboratory conditions.

AGE-STRENGTH RELATIONSHIPS

Relationships of strength to age for the different cements, derived from Table I, are of interest in comparing rates of strength development. It might be expected that the type II cements would be readily distinguishable from the type

I's in this respect, but such was not the case. Two of the type II cements, Nos. 7 and 8, exhibited relatively low strengths at 7 days with only slightly more than average increases thereafter. Nos. 9 and 14, although comparable with the type I cements at 7 days, showed higher than average increases thereafter. Cement No. 12, which was nominally type I, produced a greater percentage strength increase between 28 and 91 days than any of the others, including type II.

Given below are relative strengths of each cement expressed in terms of 28-day values:

Source	Strength, per cent of 28-day, at	
	7 days	91 days
1	67	118
2	69	110
3	75	108
4	70	110
5	73	116
6	73	106
7[a]	54	116
8[a]	59	120
9[a]	68	122
10	67	119
11	66	114
12	67	133
13	70	110
14[a]	66	126
Control Portion A	75	109
Control Portion B	75	110

[a] Type II cement.

CONCLUSION

This research confirmed the indications of earlier work involving a smaller number of cement sources. The following conclusions seem justified:

1. Average strengths for the different cements varied substantially. The range for ten type I cements at 28 days was 36 per cent of the median strength; the four type II cements were distributed over a range of 25 per cent.

2. At least one-half of the individual cements differed from shipment to shipment sufficiently to have a significant effect on uniformity of concrete strength. Half of the sources, including three of the four type II cements, yielded coefficients of variation greater than 7 per cent. Two cements had coefficients of variation greater than 10 per cent for 28-day tests. For this latter degree of variability, strength tests would be expected to drop more than 18 per cent below average about one in 20 times.

3. Mixing water requirements of mortar also varied from shipment to shipment as well as between sources. There was no conclusive evidence that these differences were related to the variability of strength except for source 12 where high water requirement tended to be accompanied by low strength. Considering indications of earlier work correlating concrete with mortar, it is doubtful that the variations in mixing water shown here would be reflected to a measurable degree in the consistency of concrete.

4. Mortar air content produced by the cements varied from shipment to shipment and source to source. The variation, with the exception of a single sample, was below the maximum limit of 12 per cent permitted by ASTM specifications for non-air-entraining cements. Within-source differences in air did not appear to correlate with strength variations.

5. A trend toward progressive reduction in air content of mortar from beginning to end of the program was discernible for both the control cement and the outside sources. That reduction was accompanied by increased mortar strength consistent with the changes in amount of air. The reason for these trends has not been established but may lie with the presence of small amounts of air-entraining contaminants in the standard sand. Analyses indicate that the strength comparisons and conclusions regarding uniformity were not vitiated by the air content trends.

6. Tests of multiple samples from two cars of cement showed them to be highly uniform throughout. In fact, differences in results of these within-car tests could all be attributed to normal testing variation, which would have occurred even if all of the samples had been absolutely identical.

7. Parallel tests by two operators yielded essentially identical mortar strength levels and uniformity. There was a consistent slight difference in weights of cubes, possibly caused by effects of compactive effort or accuracy of strike-off.

Acknowledgments:

The tests were performed at the Joint Research Laboratory sponsored by the National Sand and Gravel Assn. and the National Ready Mixed Concrete Assn. at the University of Maryland. The project was approved by the Joint Research Committee of the two associations on the recommendation of the Cement Committee of the NRMCA. It was carried out under the supervision of Richard D. Gaynor, Associate Research Engineer and Laboratory Manager, and under the general direction of the authors.

Mr. Gaynor assembled the data and prepared two progress reports.[6] John R. Wilson, Associate Engineer, contributed substantially in summarizing and analyzing the data. Herman Knoppel, Jr., Assistant Laboratory Manager and Senior Technician, and Arthur C. Ellis, Laboratory Technician, molded the specimens and made the strength tests.

Especial thanks are due to the member companies who cooperated so efficiently in securing, packaging, and shipping the samples on time. They are not named in the interest of maintaining the anonymity of the cement sources.

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[6] "Uniformity of Strength Producing Properties of Portland Cement," Feb., 1960 and "Studies of Variations in Portland Cement," Jan., 1961, special reports, National Ready Mixed Concrete Assn.

THE MECHANISM OF GRINDING AIDS*

BY F. J. MARDULIER[1]

SYNOPSIS

Grinding aids, interground with portland-cement clinker at relatively low addition rates, are generally polar compounds. Because of their polar nature, such compounds are preferentially adsorbed on reactivity centers of cement surfaces formed probably by fracture of electrovalent bonds and thus reduce the surface energy forces which cause agglomeration of the newly produced cement particles. The consequent reduction in surface energy forces causes "dry" dispersion of the cement which, in turn, increases cement fluidity and in some cases significantly reduces mill retention time. To aid in the control of mill retention time, a method for its determination involving the use of fluorescein has been developed. The relationship between mill retention time, circulating load, and ratio of grinding media to clinker charge is shown. An approach to the determination of optimum circulating load is outlined.

Grinding aids may be described as chemical additions, interground in amounts usually less than 0.25 per cent, which either increase grinding mill production rates at a given fineness or increase fineness at the same production. Grinding aids for portland cement are subject to the further qualification that they shall have no detrimental effect on the finished product. Criteria for determining the non-detrimental character of grinding aids are provided in the proposed ASTM Specification for Processing Additions for Use in the Manufacture of Portland Cements.[2] A brief history of the development and acceptance of cement grinding aids has been given by Blanks and Kenedy (**1**).[3]

The available literature indicates that grinding aids function to (1) reduce agglomeration of comminuted particles, (2) reduce surface tension at the solid-air interface of the particles being ground, and (3) reduce or eliminate coating of the grinding media. The purpose of this paper is to present evidence in support of the first of these functions with only brief comment on the latter two.

When grinding aids began to be used commercially in the 1930's, dusting was commonly observed in mill rooms where dust collection facilities were inadequate. The atmosphere of the mill room became hazy because of the presence of extremely fine cement particles suspended in the air. Since dusting did not occur when a grinding aid was not in use, this phenomenon was attributed to "dry" dispersion of the cement by the grinding aid during and after grinding (**2**). Fine particles which normally would adhere to coarser

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* Presented at the Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

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[2] ASTM Specification for Processing Additions for Use in the Manufacture of Portland Cement (C 465 - 61 T), 1961 Book of ASTM Standards, Part 4.

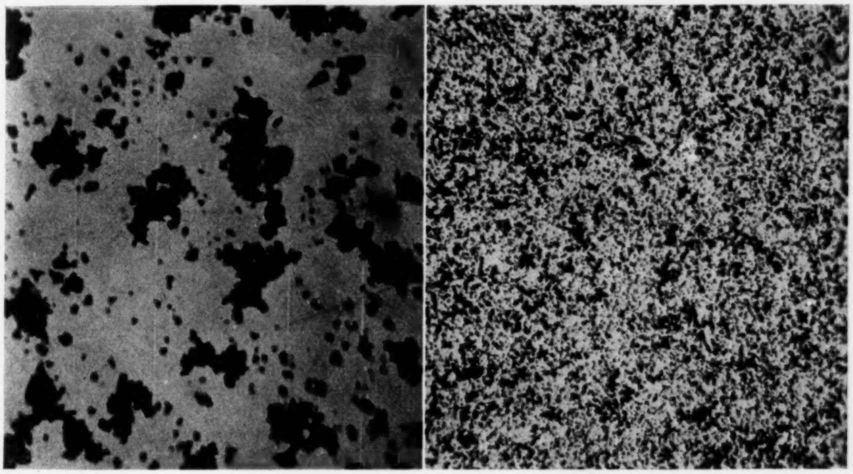
[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

particles or to groups of other fine particles to form flocs or agglomerates were freed by dry dispersion to act as separate entities and, as a result, escaped from the grinding circuit.

In some instances, the addition of a grinding aid to a preliminary grinder has been found to cause the partially ground cement in the secondary grinding mill feed bin to spurt through small holes in the bins or to flush through the clinker feed screws which normally were adequate for controlling the tube mill feed rate. In one extreme case, the preliminarily ground clinker spiralled through its feed screw so rapidly that the mill became filled, ball cascading ceased and mill power consumption dropped to only 10 per cent of its normal figure.

These and similar observations over a 25-yr period confirmed the hypothesis that grinding aids generally function by dry dispersing the clinker being ground, that is, by reducing significantly the forces of interparticle attraction which usually caused repeated agglomeration of the fine particles produced during grinding. These observations also established that grinding aids could be effective even when ball coating did not exist and indicated that ball coating might be a special and extreme case of agglomeration.

The existence of cement agglomerates is demonstrated by Fig. 1(*a*) showing a cement containing no grinding aid and Fig. 1(*b*) showing the same clinker ground with a grinding aid. These photomicrographs were taken with transmitted light at 700 magnification of small samples of dry cement vibrated on a glass slide. Although agglomerates are still apparent in Fig. 1(*b*), they are much smaller in size and clearly show the reduction in the tendency of the treated cement particles to stick together.



(*a*) Agglomerated. (*b*) Dry dispersed.

FIG. 1.—Small Samples of Dry Cement Vibrated on Glass Slide and Viewed Microscopically with Transmitted Light at 700 Magnification.

The development of agglomerates during grinding has been demonstrated by Papadakis (3) who deflocculated freshly ground cement samples by a fluid agitating technique which substantially increased the Blaine surface area of these samples without subjecting them to further grinding. The greater specific surfaces thus obtained showed compliance with Rittinger's law in the range of Blaine surface areas of 0 to 6000 sq cm per g. Such compliance is not normally found in the upper two-thirds of this surface area range.

CAUSES OF AGGLOMERATION

The forces causing agglomeration during grinding have been attributed by some investigators to static electrical charges of opposite polarity (**4**). The existence of static electrical charges on cement particles under low humidity conditions has been noted by the author. However, in every instance these charges gave evidence of having the same polarity, thus causing the particles to be repelled from each other and to stick to a dissimilar surface bearing the opposite charge. Hence, static electrical charges developed by friction during grinding do not appear to constitute the forces of interparticle attraction which cause agglomeration during grinding. Scott (**5**), after laboratory investigation, concluded that the forces of interparticle attraction causing pack set likewise were not due to static electrical charges.

TABLE I.—GRINDING AID EFFECT OF AN ANTI-STATIC COMPOUND.

	Blank—No Grinding Aid	Reference Grinding Aid[a]	Anti-Static Compound
Addition rate, per cent by weight solids	0	0.03	0.03
Mill revolutions[b]	2500	2500	2500
Blaine surface area, sq cm per g[c]	4325	4840	4755
Surface area per mill revolution	1.73	1.94	1.90
Ratio, per cent	100	112	110

[a] Lignin-type grinding aid.

[b] Two individual grinds for each condition of test on different days. Mill temperature 73.5 F.

[c] Each value an average of 6 determinations on three different samples.

Confirmation of the conclusion regarding grinding is given in Table I, which shows the grinding aid effect of an anti-static polar compound whose function was to provide a conductive path by which static charges developed on the clinker particles during grinding could travel to the grounded shell of the mill. If agglomeration during grinding is partly or entirely due to the attractive forces of static charges of opposite polarity, anti-static agents should be superior grinding aids. These data show the one tested to be no better than would be expected for any similar polar grinding aid, and in fact slightly inferior to the control grinding aid having no de-staticizing properties. In addition, this agent was an air-entraining agent, which fact renders it unusable as a processing addition.

Static electrical charges of like polarity seem to be developed quite readily on particles of clinker or cement under certain dry atmospheric conditions by frictional contact among the particles or between particles and grinding media, mill lining plates, steel-pipe transfer lines, and the like. Such similarly charged particles will repel each other. Due to unusual accumulation of free electrons under certain favorable conditions, the static potential on cement particles may in some cases become great enough to cause an observable discharge across a small air gap to a suitable ground. In measuring the static charges formed on cement particles by grinding or similar means, great care must be taken not to induce static charges on the cement particles by exposing them to a static electrical field (**5**). Such exposure can cause a redistribution of free electrons on particle surfaces, which, in turn, may cause a particle to act like a charged particle, that is, one which has either gained or lost electrons. Static electrical charges are known to exist on exterior surfaces only; their surface densities (but not their polarities) depend upon surface convexity.

Some investigators have attributed agglomeration of cement particles to the almost instantaneous growth of tiny crystals common to adjacent particles. Bikerman (6) has stated that this is possible even when moisture content is as low as 0.01 per cent of the weight of the particles. Many field experiences and observations pertaining to grinding, grinding aids, and pack set appear to be explainable on the basis of this hypothesis. It seems reasonable that cement particles under the conditions of temperature and pressure existing in silo storage might well agglomerate due to crystal growth, and this may be a factor in "silo set" of cement in storage which has also been considered as an exaggerated case of pack set (7). However, since crystal growth would be more likely at high moisture contents than low, this hypothesis does not appear to explain the action of water as a grinding aid and as a pack set inhibitor in some cases.

A third explanation of particle agglomeration during grinding involves surface energy forces which exist on all surfaces and whose intensities depend upon their nature and the degree to which they are counteracted by externally applied forces or screens. Discussion of this phase follows.

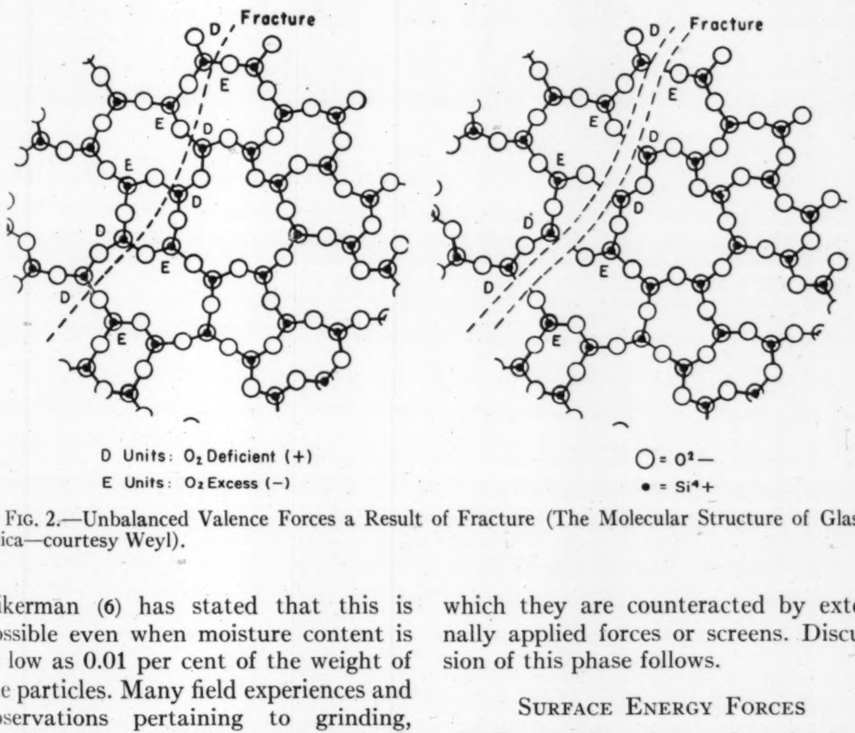


FIG. 2.—Unbalanced Valence Forces a Result of Fracture (The Molecular Structure of Glassy Silica—courtesy Weyl).

SURFACE ENERGY FORCES

Jeffery (8) has shown that the basic building block in the molecular structure of C_3S and C_2S is the $SiO_4^{\equiv}$ tetrahedron of silica. Van Vlack (9) has explained how the metallic Ca^{++} ions are incorporated in the molecular structure of silica to form calcium silicate. The fact that silica (SiO_4) is the hardest of the three compounds to grind and that grindability of these compounds increases with the number of Ca ions included in the molecular structure is probably significant with regard to structural density and defects. Since the lime silicates comprise about 80 per cent of portland cement composition, it seems likely that deductions concerning surface energy development can be based on the simpler molecular structure of silica.

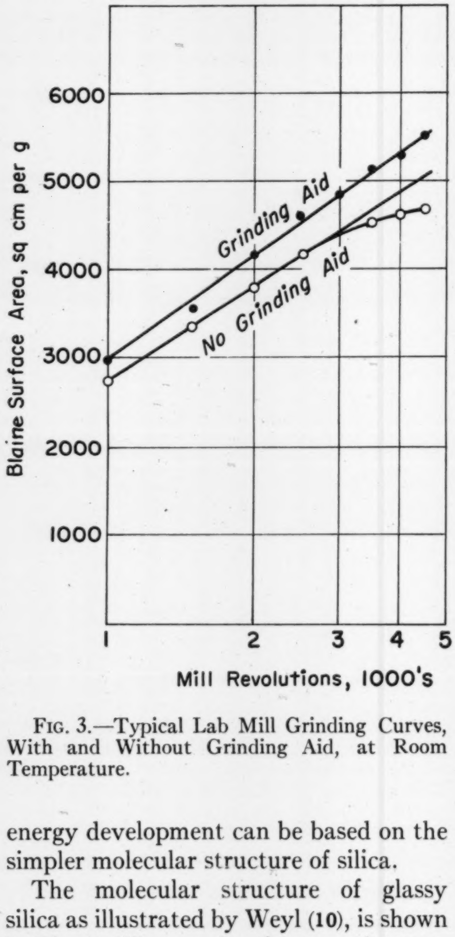


FIG. 3.—Typical Lab Mill Grinding Curves, With and Without Grinding Aid, at Room Temperature.

The molecular structure of glassy silica as illustrated by Weyl (10), is shown in Fig. 2. When such a framework structure is fractured, the fracture line severs electrovalent bonds as shown, causing unbalanced primary valence forces of opposite polarity to exist on each of the two surfaces created by the fracture. Because of this severance of electrovalent bonds, Gaudin (11) has likened a grinding operation to a chemical reaction. Gaudin has further pointed out that were it possible to fit together again precisely two such newly-formed surfaces before any of the unbalanced valence forces have been partially or completely satisfied by adsorbed foreign ions, the original electrovalent bonds holding these two surfaces together would be re-established. The particle which had been fractured would become whole again!

That this analogy applies to portland-cement clinker despite its different composition has been indicated by Hansen (12) who views the surface of a particle of cement as containing a series of alternate Ca^{++} and $O^{=}$ reaction sites. The absence of corresponding Si reaction sites is explained by Weyl's work which indicates that the Si cations have probably retracted from the surface in a molecular structural adjustment designed to produce minimum surface energy and, therefore, maximum surface stability. These reaction sites, regardless of their identity, appear to coincide with the surface energy centers; that is, both result from severance of the corresponding electrovalent bonds. Possibly the non-siliceous interstitial material in portland-cement clinker similarly contributes to the surface energy of the ground cement particles.

In actual practice, it is impossible to fit together such newly formed surfaces precisely and instantaneously after fracture. However, the action of the grinding media in a revolving mill not only fractures existing clinker particles, but also violently compresses groups of particles already formed, causing approximate fitting of some of the residual unbalanced valence forces of opposite polarity. Thus, agglomerates are formed. There is field evidence to show that with mills of large diameter having short mill retention times some of these valence forces may not be completely satisfied even for some time after the cement leaves the mill.

The compression of groups of particles is statistically more likely to occur with fine particles than with coarse and with large diameter grinding media than small. In addition, the surface energy forces are a direct function of surface area produced. Thus agglomeration is greater the finer the cement being ground. This conclusion is substantiated by increasing spread between the tangents to the two typical laboratory mill grinding curves shown in Fig. 3, the deviations from tangents for the lower curve indicating increasing development of ball coating.

TABLE II.—EFFECT OF TYPE OF GYPSUM ON CLINKER GRINDABILITY, WITH AND WITHOUT GRINDING AID.

Gypsum	Without Grinding Aid			With Grinding Aid		
	Rev	B-SA[a]	SA/Rev[b]	Rev	B-SA[a]	SA/Rev[b]
A	3500	4980	1.425	2400	4940	2.060
B	3200	5080	1.585	2500	4980	1.995
C	3900	4940	1.270	2600	5050	1.950
D	3700	4940	1.335	2600	5050	1.950
Avg	3575	4985	1.404	2525	4995	1.990

[a] Blaine surface area.

[b] Surface area per revolution.

Gypsum, one of the essential ingredients in portland cement, is relatively easier to grind than clinker; hence, it is the first ingredient to be reduced during grinding to a fineness at which strong agglomerating tendencies exist. Possibly for this reason or static electrical attraction of gypsum to steel surfaces, chemical analyses reveal that gypsum predominates in ball coating. Laboratory studies by Bond and Agthe (**13**) show less ball coating when grinding a given clinker without gypsum to the same No. 325 fineness than obtained with gypsum interground. The fact that the corresponding Wagner surface area was substantially less for the cement without gypsum shows a lack of fines when no gypsum was present and tends to confirm that fineness is the predominant factor causing ball coating. However, ball coating does develop without gypsum when the clinker is ground fine enough.

The effect of gypsum fineness on surface energy forces and ball coating is complicated by the variable effect of different gypsums on grindability shown in Table II, in which relative grindability is expressed as Blaine surface area units per laboratory mill revolution. All gypsums were interground in such amounts as to give 2.4 per cent SO_3 in the finished cement. Gypsum C was a synthetic by-product gypsum. The range of grindability factors shown is approximately 22 per cent of the average for the grinds made without grinding aids. Possible reasons for these variations are discussed later.

ACTION OF GRINDING AIDS

Grinding aids appear to function by coating the cement particles, thus "shielding" the forces of interparticle attraction which cause agglomeration. Based on the surface energy hypothesis just proposed, it is technically more accurate to say that the grinding aid provides readily available foreign ions to satisfy the unsatisfied valence forces produced by fracture of the clinker during grinding.

Experience shows that the most effective grinding aids are those which are polar in nature. Polar compounds, because of their asymmetric structure, have centers of positive and negative charge which are not coincident and hence develop a "dipole" moment in a force field which causes the dipole to orient itself with the direction of the force field. For this reason, polar compounds are preferrentially adsorbed at the unbalanced valence force sites as the new surfaces are formed and thus reduce agglomerating tendency more effectively than non-polar compounds.

Since polar compounds are attracted to the specific sites where the surface energy forces exist, lesser quantities of additive are required to do an effective shielding job than when non-polar compounds are used. As little as 0.01 per cent by weight of cement of certain grinding aids has been found highly effective, although theoretical calculations based on molecular weight of the grinding aid and the Blaine surface area of the cement indicate that ten times as much may be needed to "coat" the entire surface area of the cement with a monomolecular film.

The amount of additive required to obtain effective grinding aid is important because of the requirements that (1) the use cost of the grinding aid be realistic and (2) there be minimum interference by the adsorbed grinding aid with the normal hydration process of the cement. The composition of the grinding aid can markedly affect early hydrolysis and hydration at even relatively low addition rates. This is clearly demonstrated by the use of organic retarders, either admixed or interground, to supplement gypsum where further delays in set are needed to maintain workability over longer periods of time. All of the additives of this type discussed during the ASTM Symposium on Water-Reducing Retarders (San Francisco, 1959), as well as those reported in the February, 1961, Issue of *Public Roads* were polar compounds (**14,15**).

Ziegler (**16**) has clearly demonstrated the superior grinding aid effectiveness of polar compounds by making closely controlled laboratory grinds with vacuumized mill. His procedure made possible the substitution of various polar and non-polar vapors for dry air as the grinding mill atmosphere. Dipole moment, compound volatility, and possibly dielectric constant are indicated as being factors in the greater effectiveness of the polar compounds. Ziegler independently obtained photomicrographs similar to those of Fig. 1. He attributes grinding aid effect to reduction in surface tension at the solid-vapor interface as well as to reduced agglomerating tendency.

Water is a polar compound which has been recognized as a grinding aid for many years and whose grinding aid effectiveness is limited by its low dipole moment and low molecular weight. Its shielding effect is not great. To prevent detrimental effects on the cement quality and warehousing properties, its addition rate is usually limited to that which will not significantly raise the ignition loss of the resulting cement. Water is particularly effective when used as a carrier to assure proper distribution of high-dipole-moment high-molecular-weight organic grinding aids, but there have been cases when water, as a carrier, has decreased the effectiveness of organic grinding aids by causing coating of the mill lining plates.

Gypsum C which gave the poorest grindability of the four covered by Table II was a synthetic by-product gypsum containing no water of crystallization. The low grindability may be due to the lack of combined water for partial release during grinding to aid in satisfying the surface energy forces created by fracture. The effect of the natural grindability characteristics of the gypsum itself on gypsum fineness appears to be slight, based on the relatively constant grindability of the clinker with the four different gypsums when a grinding aid was used. This indicated effect of the combined water in gypsum acting to satisfy the unbalanced valence forces formed during grinding is substantiated in part by one cement company which substituted gypsum anhydrite for natural gypsum and found that mill grinding rate was reduced 6 per cent as a result (**17**). For combined water in the gypsum to perform this function it must be released as water vapor. Normally the instantaneous temperatures at grinding media point-of-impact are sufficiently high (about 500 F) to cause some of the combined water to be so released.

The use of a grinding aid with the various gypsums involved in Table II increased the average grindability factor 42 per cent, from 1.404 to 1.990 sq cm per g per rev. At the same time, the variable effect of the gypsum, whether due to water content or inherent grindability or both, was reduced to a minimum. Range of deviations from the average grindability factor with a grinding aid was only 5 per cent, from 1.950 to 2.060 sq cm per g per rev., in contrast to 22 per cent without a grinding aid.

A careful scrutiny of Fig. 1 will suggest the probable effects of grinding aids and dry dispersion on closed circuit grinding. By preventing or minimizing agglomeration, air separator rejects are cleaner, that is, contain fewer acceptable fines, and thus fewer fines are returned to the mill for regrinding or, in some cases, overgrinding. As a result, air classifier operation is improved, and the quantity of "super fines" in the final product is reduced. The latter can be desirable or not depending upon the particle size distribution of the cement initially and the collateral effects of dry dispersion on mill retention time. The adsorption process by which grinding aids attach themselves to particles of cement is clearly demonstrated by the fact that chemical analyses of air separator fines, feed, and tails show their grinding aid content to be proportional to surface area when the mill and separator are in equilibrium. The same proportionality does not always exist when the grinding aid is added as a dry powder rather than a water solution.

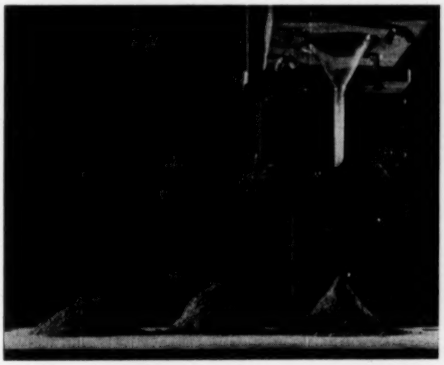


FIG. 4.—Angle of Repose of Treated and Untreated Cements.

EFFECT ON MILL RETENTION TIME

Dry dispersion causes cement to be less sticky and, therefore, more free-flowing after grinding (**18**), as well as during grinding, as shown by the angles of repose of the three piles of cement shown in Fig. 4. The two cements on the left were ground in a laboratory mill at 235 F with two different pack set inhibitors and the cement on the right contained no processing addition. The individual piles were formed under constant and comparable conditions by using the vibrating funnel shown in the upper part of the photograph. However, in the laboratory grinds, the cement on the left, which shows the smallest angle of repose, had the same grindability as that in the center pile, showing that the two processing additions used were equally effective grinding aids, although unequal in pack set inhibition. This fact is significant with regard to the relative intensity of the surface energy forces causing agglomeration during and after grinding.

The severance of electrovalent bonds by fracture of clinker particles during grinding results in primary valence forces of maximum intensity being created on the new surfaces at the instant of fracture. Immediately after fracture, however, these intense forces of attraction begin adsorbing foreign ions from the mill atmosphere or from grinding aids when used. Some of them may also become coated by non-polar contaminants like lubricating oil or establish relatively weak bonds with their valence opposites on other particles thus causing agglomerates. By so doing, the surface energy level of the comminuted particles within the mill is rapidly reduced, but tests measuring quantitatively the pack set or agglomerating tendency of cement after grinding show that this process of reduction of surface energy to its natural minimum usually continues at a lower rate for several days or weeks after the cement leaves the mill. For this reason, aged cement has less pack set tendency than freshly ground cement at constant temperature. Degree of aeration after grinding affects the speed at which minimum surface energy levels are reached.

TABLE III.—REDUCTION IN PACK SET TENDENCIES OF TWO CEMENTS.

Cement	Grinding Aid, per cent[a]	Blain Surface Area	Pack Set Index
A	0	2905	46
A	0.033	3130	8
C	0	3055	15
C	0.033	3140	7

[a] Amount of grinding aid—solids by weight of cement.

It seems axiomatic that the rate of reduction of the forces causing agglomeration resulting from foreign ion adsorption will be greater at high energy levels than at low energy levels. Table III gives pack set index data confirming this conclusion (**19**). Because cement C had a low pack set index initially, the addition of a polar compound caused only a 2:1 reduction in agglomerating tendency as against a 6:1 reduction for cement A. By extrapolation from these data obtained subsequent to grinding, it can be deduced that during grinding the reduction in agglomerating tendency due to the addition of a grinding aid will be considerably greater. It is also interesting to note that there appears to be an irreducible minimum surface energy level represented by the pack set indexes of 7 and 8 for treated cements C and A, respectively.

It can be concluded from these data that flowability of the partially ground clinker in the mill may be increased substantially by intergrinding a processing addition. Just as reduction in viscosity by means of an additive will increase rate of open flow of a heavy oil in a gently inclined tube, so flow of cement through a mill may be increased even though total feed to the mill is held constant. Thus dry dispersion of cement may and frequently does affect mill retention time, all other factors being constant, and may, in turn, affect mill operating efficiency and particle size distribution of the finished product.

Mill retention times have been determined using a new procedure specifically designed to determine the effect of grinding aids on the rate at which cement particles on which the grinding aid is adsorbed move through the grinding unit. The method involves the use of fluorescein as a tag or tracer additive. Fluorescein for this purpose is unique for its (1) tremendous spreading power, (2) resistance to the alkalinity of the cement, (3) ready solubility in water used to extract it from cement, and (4) ease of quantitative determination by means of inexpensive fluorescence measuring equipment capable of detecting as little as 10 ppb by weight of the cement.

A predetermined quantity of fluorescein solution is added to the clinker entering the grinding mill; samples of the mill discharge cement are taken at appropriate intervals thereafter, and then the fluorescein content of the grab samples so taken is determined. A plot of fluorescein content against sampling time for an 11½ by 17-ft. mill close circuited with an air separator is given in Fig. 5.

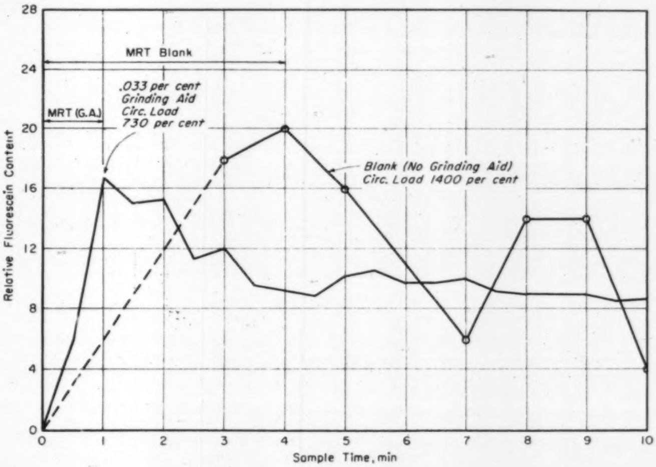


FIG. 5.—Typical Mill Retention Time (MRT) Curve for 11½ by 17-ft Mill, With and Without Grinding Aid.

The astonishing reduction in mill retention time (MRT) from 4 min to 1 min, resulting from the addition of a grinding aid, not only shows the unusually great dry-dispersing effect of the addition on this particular cement, but warns that certain adjustments must be made to control mill retention time if commensturate grinding aid benefits are to be realized. It is a matter of record that a commercial grinding aid was, in one instance, found to perform as a grinding inhibitor, probably because of excessive reduction of retention time. Contrary to normal expectations, its use caused a reduction in mill discharge fineness and an increase in circulating load. The remedy which was to reduce the grinding aid addition rate by 50 per cent was simple and effective, but was not conducive to getting maximum savings from the use of the grinding aid; in fact, abnormally high production rates might have been obtained had other means been used to control retention time.

When mill retention time in open circuit grinding is markedly shortened by dry dispersion, the reduction in agglomerate formation may be more than compensated for by the reduced grinding time, and a decrease in fineness or production rate will result. If these two tendencies more or less balance, little measurable change in fineness or production rate will result. If the same surface area is obtained with a substantial shortening of mill retention time, particle size distribution may be changed as shown in Fig. 6.

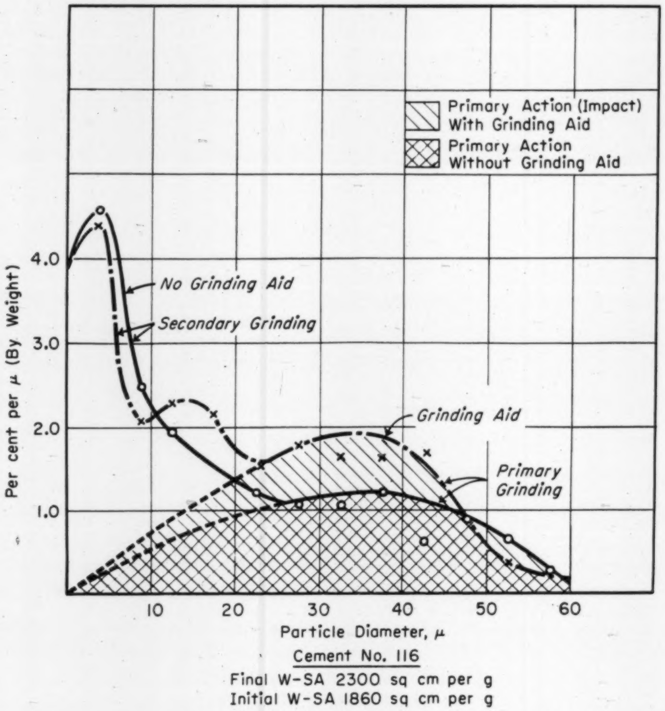


FIG. 6.—Effect of Mill Retention Time on Particle Size Distribution.

The two cements for which the size distribution curves are shown in Fig. 6 were ground with and without a grinding aid to the same Wagner surface area in a laboratory batch mill. Grinding time was shorter with the grinding aid. Wagner particle size distributions for the two cements were calculated in terms of percentages of particles of various sizes per unit of diameter (microns) to demonstrate more clearly the primary (impact) and secondary (attritional) actions involved. These data show that the grinding aid increased impact grinding and decreased attritional grinding. Since attritional grinding is inefficient because the abrading action involved dissipates more of the input energy in the form of heat, this change in the ratio of impact to attritional grinding may explain part of the improved mill efficiency obtained with a grinding aid.

CLOSED CIRCUIT GRINDING

The improvement of separator efficiency through dry dispersion obtained with a grinding aid has already been mentioned. The shortening of mill retention time through dry dispersion may impair mill efficiency by reducing the volume of cement in the mill to the point where the mill is starving. Under such conditions, there is increase in some grinding of steel on steel, mill temperatures rise, and production rate per unit of power used is reduced. This may occur at identical calculated circulating load percentages with normal or larger than normal feed rates to the mill. A corrective is to increase the per cent circulating load, but this is seldom possible since most closed circuit units are always operated at full capacity of the circulating load system when no grinding aid is used.

However, such a corrective does not get at the core of the problem and may lead to other inefficiencies. For example, in the exceptional case illustrated by Fig. 5, total retention time with the grinding aid was approximately 8 minutes, total retention time being the mill retention time for a single pass through the mill multiplied by the number of passes indicated by the corresponding circulating load. The total retention time without the grinding aid was approximately 60 minutes. To obtain a 60-min total retention time with the grinding aid, a circulating load of more than 10,000 per cent would be required. Such a circulating load would not only be impractical but would (1) increase substantially the power consumption per unit of new clinker feed, (2) further reduce mill retention time, and (3) probably cause flushing of unground clinker through the mill. Lengthening the mill retention time by mechanical adjustment of the mill is the only practical solution in such a case.

With a short mill retention time and insufficient circulating load capacity, it is likely that a mill will "starve," that is, the ratio of volume of grinding media to volume of clinker being ground will be excessive. The ideal ratio of weight of grinding media to weight of clinker being ground has been shown by various investigators to range from 7:1 to 30:1, depending upon many test factors. The current consensus seems to be that 9:1 is a good average, but this will obviously vary with local conditions. It is believed that maximum production rate per unit of power consumed will be obtained with the ideal ratio.

Knowledge of the mill retention time makes it possible to determine the amount of partially ground clinker in a mill at any instant. Knowing the weight of grinding media involved, the ratio of grinding media weight to weight of clinker being ground can be calculated using the following equation:

$$R = \frac{15.95W}{TP(L + 100)} \quad \text{..........} (1)$$

where:

W = weight of grinding media, lb,

T = retention time, min,

P = production rate, bbl per hr,

L = circulating load, per cent, and

$$15.95 = \frac{60 \text{ (min per hr)} \times 100 \text{ (\%)}.}{376 \text{ (lb per bbl)}}$$

If R_o = optimum ratio of grinding media weight to weight of clinker, the optimum circulating load, L_o, for a given mill retention time and production rate can be calculated as follows:

$$L_o = \frac{15.95W}{R_oTP} - 100 \quad \text{........} (2)$$

From this equation, it is clear that optimum circulating load for an optimum ratio of grinding media weight to clinker weight depends upon mill retention time to a considerable extent, since retention time can vary over a large range, and on production rate which for a given fineness varies over a small range. Because the circulating load cannot be changed without at the same time changing the feed rate of new clinker to the mill and affecting mill retention time, control of circulating load is of little help in maintaining the ideal ratio of grinding media to clinker. Mechanical control of mill retention time offers a useful method of accomplishing this objective, thus improving mill efficiency and particle size distribution control.

CONCLUSIONS

The foregoing discussion of the mechanism of grinding aids should be helpful in understanding how grinding aids function and how to get the most out of their use. The author believes that it is just as necessary to adjust a grinding mill mechanically to control mill retention time and assure optimum, or at least constant ratio of weight of grinding media to weight of clinker, as it is to adjust an automobile carburetor to the octane rating of the gasoline used so that maximum savings in gasoline consumption may be realized.

Only in this way can maximum mill efficiency through the use of a grinding aid be assured. Such control should be mechanical through the adjustment of screen plate openings, increase in density of ball loading or elimination of discharge screen plates. Reduction in the amount of grinding aid used sometimes helps increase mill retention time but seldom gives optimum results because agglomeration is thereby only partially reduced. Control of the ratio of grinding media weight to clinker weight by means of controlling circulating load is not possible, since retention time varies inversely as the circulating load.

It is probable that much can be gained through the determination of mill retention time and ratio of grinding media weight to clinker weight. Not only will grinding aid effectiveness be improved, but product quality uniformity may be enhanced by better control of particle size distribution of cement during grinding.

Acknowledgment:

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DISCUSSION

MR. W. C. HANSEN[1] (*presented in written form*).—Mr. Mardulier is to be congratulated on clarifying the mechanism by which grinding aids function in the grinding of portland cement. This discussion is an attempt to furnish additional information regarding the conclusions of the author.

Weyl[2] points out that the surfaces of particles of MgO in powdered periclase do not contain the expected number of Mg^{++} ions but consist primarily of polarized $O^{=}$ ions because the strong potential field of the small and weakly polarizable Mg^{++} ion raises the surface energy much more than that of the large and polarizable $O^{=}$ ion. Accordingly, the particles of MgO repel each other because of the excess negative charges carried by their surfaces.

In the case of $3\ CaO \cdot SiO_2$, the crystals contain four positive Ca^{++} and Si^{4+} ions to five negative $O^{=}$ ions. In $3\ CaO \cdot Al_2O_3$ there are five positive Ca^{++} and Al^{3+} ions to six negative ions. Even though the positive ions are less polarizable than the $O^{=}$ ions, they probably are numerous enough to give particles with surfaces that contain about equal amounts of positive and negative charges. Hence particles of portland cement should not repel each other to any great extent and, as the author points out, might readily form agglomerates under the impact of the balls during grinding.

The author points out that the efficiency of a compound as a grinding aid is a function of its dipole moment. In this connection, Lewis[3] presents evidence to show that the deflocculation of Al_2O_3 particles in suspensions is not compatible with the double-layer theory for the deflocculation of suspensions but is compatible with a polar-screening theory. Alumina particles are similar to those of MgO in which the small weakly polarized cation is screened by the highly polarized $O^=$ ion to give particles with surfaces that are negatively charged.

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[1] Consulting Chemist, Valparaiso, Ind.

[2] W. A. Weyl, "A New Approach to Surface Chemistry and Heterogeneous Catalysis," *Bulletin No. 57*, Mineral Industries Experiment Station, Pennsylvania State College, State College, Pa.

[3] Arthur E. Lewis, "Polar-Screen Theory for the Deflocculation of Suspensions," *Journal*, Am. Ceramic Soc., Vol. 44, pp. 233–239 (1961).

In distilled water the negative charge of the Al_2O_3 particle is neutralized by the adsorption of H_3O^+ ions and the particles are flocculated. When a base is added to the water, the H_3O^+ ions are removed and the suspension can be deflocculated by the adsorption of polar ions or polar molecules. In this case, there appeared to be no deflocculation except by ions or molecules capable of hydrogen bonding with the $O^=$ ions in the surface of the grain of Al_2O_3. The greatest success was obtained with organic acids capable of strong hydrogen bonding. A much weaker deflocculation was obtained with compounds capable of weak hydrogen bonding, such as glycerin and sugar.

In order to demonstrate the incompatibility of the double-layer theory, Lewis carried out experiments with Al_2O_3 suspensions in benzene, a nonpolar liquid. Various organic acids were added to the benzene, and since they would not ionize in this solvent, their deflocculating ability must have been the result of the adsorption of the polar molecule by hydrogen-bonding and not by the adsorption of a charged ion. Acids with dipole moments were effective deflocculants, whereas those with zero dipole moments were not. Lewis states "The failure of the acids with zero dipole moment to produce deflocculation is expected because all the acids with zero dipole moment tested have a symmetrical distribution of COOH groups that permits strong hydrogen bonding of the groups to each other."

It seems that there might be a marked similarity between a suspension exposed to a polar compound in a solvent such as benzene and a powder interground in a ball mill with a polar compound. It appears, therefore, that the work by Lewis supports the conclusions of the author with respect to the type of surface forces involved in the grinding of portland cement and the mechanism by which the agglomerates are deflocculated.

The author points out that anhydrous calcium sulfate is very ineffective as a grinding aid, whereas gypsum appears to act to some extent as a grinding aid. This has been explained on the basis that the latter liberates water during grinding and that this water produces a conducting atmosphere. It would seem that gypsum might be capable of hydrogen bonding with the cement particles and in that way function as a grinding aid. The author suggests that water is adsorbed as a dipole molecule and is a mild grinding aid. It seems that the adsorption of the gypsum by hydrogen bonding might be superior from the standpoint of the grinding aid effect than the adsorption of the same quantity of water. Adsorbed water might migrate into the crystals and have very little effect in changing the charge on the surface of the crystal.

These papers by Mardulier and Lewis bring to mind a statement made by Bello[4] in discussing an experiment in biology. He stated "Like many of the great experiments in science, it showed that what everyone believed to be so was not so." It has generally been believed that the agglomerating forces involved in grinding were static charges. Likewise, one hears statements to the effect that cement suspensions show a zeta potential because of double-layer adsorption. The work of Lewis on suspensions of alumina suggests that this might not be true.

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[4] Francis Bello, "Great American Scientists; The Biologists," *Fortune*, June, 1960, p. 229.

MR. F. J. MARDULIER (*author's closure by letter*).—Mr. Hansen's discussion, showing that investigators in other fields have obtained results tending to support the hypothesis presented in "Mechanism of Grinding Aids," is much appreciated.

There seems to be complete agreement on the essential item that surface energy forces cause agglomeration during grinding. However, the precise mechanism by which grinding aids are adsorbed by particle surfaces and thus reduce the surface energy forces causing agglomeration remains somewhat in doubt. Dipole moment of the grinding aid is a major factor but not the only one. Also, the rate at which minimal surface energies are reached as a result of the adsorption of foreign ions and the structural adjustment of the surface molecules is still controversial.

Fortunately, final resolution of these mechanism matters is not necessary for the satisfactory application of the surface energy hypothesis to practical field problems involving grinding and pack set. The hypothesis as presented has been found to be a useful tool and guide in analyzing and improving grinding operations, and in explaining pack set phenomena.

VARIATION IN POZZOLANIC BEHAVIOR OF FLY ASHES*

By Robert D. Vincent,[1] Manuel Mateos,[2] and Donald T. Davidson[3]

Pozzolans[4] may be either natural or artificial. Natural pozzolans include some volcanic ashes, diatomaceous earth, and bauxite. Artificial pozzolans include heat-treated clays and shale, blast-furnace slag, and fly ash.

Fly ash, produced at power plants which burn pulverized coal for fuel, is collected as a by-product from stacks either by electrical or mechanical precipitators. The production of fly ash in the United States reaches 10 million tons annually. With such a great quantity produced, there is naturally much interest in finding commercial uses for it. Some of the promising and established uses for fly ash are: as a grouting component, as a mild abrasive, as a filtering medium, as an admixture in structural and mass concrete, and as a soil stabilizer (**1**).[5]

The use of fly ash in soil stabilization for roads and other kinds of pavements has been prompted by its low cost and the shortage of good natural base-course materials in many areas. Soil stabilization with lime - fly ash mixtures is still in the experimental stage, but many streets and highways have been so constructed, and recent reports indicate that most of the projects are satisfactory (**12**). Roads have been built with lime - fly ash stabilized soils in Pennsylvania, Maryland, New Jersey, and Iowa.

The Soil Research Laboratory of the Engineering Experiment Station of Iowa State University of Science and Technology has been investigating the use of lime and fly ash for soil stabilization with the assistance of the Iowa State Highway Commission and several private companies. In 1957 test sections of primary road totaling approximately 3000 ft of lime - fly ash stabilized base and 1000 ft of lime - fly ash stabilized sub-base were constructed near Colfax, Jasper County, Iowa. After four winters, these experimental sections are in excellent condition. This field trial is still under investigation and a final report will be made soon (**6**).

In investigations to correlate strength of lime - fly ash mixtures with chemical or physical properties of pozzolans, only general indications were obtained (**2,7**). However, on the basis of these and similar investigations, tentative specifications for use of pozzolans in concrete, based mostly on chemical and physical properties, have been established (**10**).[4]

More information is needed on the lime - fly ash reaction, particularly about products formed, and on the effects of chemical and physical properties, crystalline structure, and heat of reaction on strength of lime - fly ash mixtures.

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[4] 1958 Book of ASTM Standards, Part 4.

[5] The boldface numbers in parentheses refer to the list of references appended to this paper.

TABLE I.—PHYSICAL PROPERTIES OF FLY ASHES.

Fly Ash	Per Cent Passing No. 100 Sieve	Per Cent Passing No. 200 Sieve	Per Cent Passing No. 325 Sieve	Specific Gravity,[a] sq cm per g	Specific Surface[a]	Hygroscopic Moisture Content, per cent
No. 1	99.0	93.9	73.6	2.39[b]	2950[b]	0.03
No. 2	93.6	86.9	72.5	2.28[b]	4550[b]	0.04
No. 3	98.1	93.2	68.2	2.61[b]	3070[b]	0.03
No. 4	97.2	94.1	75.7	2.61[b]	2720[b]	0.03
No. 5	98.2	90.1	70.8	2.26[b]	4490[b]	0.02
No. 6	98.2	91.6	80.2	2.52	2336	0.02
No. 7	94.3	80.9	54.9	2.37	4550	0.08
No. 8	93.5	86.8	49.8	2.39	2663	0.03
No. 9	92.1	85.7	51.3	2.04	3112	0.07
No. 10	89.8	64.3	22.6	3.43	576	0.01
No. 11	98.2	91.6	31.8	2.82	1460	0.01
No. 12	90.0	72.5	54.9	2.34	4240	0.11
No. 13	53.0	24.7	14.0	2.15	1903	0.06
No. 14	95.1	82.3	64.8	2.69	2048	0.05
No. 15	93.1	75.4	57.4	2.33	1730	0.02
No. 16	98.6	86.7	57.6	2.44	2109	0.03
No. 17	99.0	94.5	82.7	2.40	3315	0.03
No. 18	99.4	94.4	86.1	2.60	3226	0.04
No. 19	93.9	81.3	66.6	2.21	2248	0.03
No. 20	91.6	73.7	47.0	2.43	3649	0.08
No. 21	96.9	84.5	80.7	2.34	2539	0.02
No. 22	90.0	70.8	53.9	2.22	1990	0.02

[a] Tests by Robert W. Hunt Co., Chicago, Ill.
[b] Data supplied by Detroit Edison Co., Detroit, Mich.

TABLE II.—CHEMICAL COMPOSITION OF FLY ASHES.

Fly Ash	Chemical Analysis, weight per cent						
	SiO_2	Fe_2O_3	Al_2O_3	CaO	MgO	SO_3	C
No. 1	41.60	25.07	24.90	2.21	2.08	0.69	6.0
No. 2	40.50	22.30	18.40	2.20	0.87	0.97	8.0
No. 3	47.00	18.40	24.00	1.65	0.84	0.55	3.5
No. 4	41.90	25.84	22.48	2.68	0.98	0.79	4.5
No. 5	57.50	6.0	22.2	1.20	0.80	0.45	4.5
No. 6	42.4	21.9	21.1	5.5	0.7	3.4	3.4
No. 7	36.20	16.68	15.80	8.3	0.92	1.47	18.55
No. 8	36.68	24.33	21.29	3.45	0.98	2.02	7.20
No. 9	43.72	18.37	16.87	6.04	1.32	1.09	8.70
No. 10	11.26	68.40	0.90	12.27	0.25	3.19	0.70
No. 11	40.09	36.65	13.13	5.80	0.30	2.36	0.15
No. 12	38.50	16.20	18.08	3.25	0.17	1.05	13.89
No. 13	28.26	22.16	9.88	8.35	0.66	1.09	28.06
No. 14	35.32	43.35	7.75	5.33	0.88	1.37	3.75
No. 15	39.16	30.22	11.92	11.60	0.77	1.86	2.85
No. 16	40.50	20.84	12.40	10.56	0.33	1.98	7.80
No. 17	45.6	17.2	21.4	6.9	1.1	2.3	4.7
No. 18	42.5	20.0	23.4	5.7	0.8	2.3	2.6
No. 19	41.6	11.9	34.7	2.1	1.3	0.8	6.9
No. 20	35.94	19.63	18.19	6.89	0.85	1.86	15.59
No. 21	51.2	20.2	10.0	6.3	1.6	1.7	1.0
No. 22	43.48	21.35	19.77	5.48	0.67	0.79	6.40

The aim of this investigation was to determine the relative effectiveness of different fly ashes for soil stabilization and also to attempt to correlate the unconfined compressive strength of compacted lime - fly ash mixtures with fly ash properties by isolating groups of fly ashes having several similar characteristics. The pozzolanic reaction of lime and fly ash was investigated with the use of unconfined compression tests, as this is a satisfactory criterion of fly ash reactivity with lime (8). To reduce variables, no soil was used and all test specimens were of commercial hydrated lime (calcitic or dolomitic), fly ash, and distilled water.

MATERIALS

Fly Ashes:

A selection was made to represent a good coverage of the major production factors believed to influence the properties and behavior of fly ashes, such as type of coal, the method of pretreatment of the coal, particle size of the coal, the type of burner, and the method of collection. The physical properties and chemical composition of the fly ashes used are shown in Tables I and II.

Descriptions of fly ash sources and their production are listed below.

Sample 1 was collected by multiple cyclone and electrical precipitators. The coal is from Districts 8 and 3 in Ohio and northern West Virginia. It is processed through pulverizing mills so that 70 per cent passes a No. 200 sieve. The sample was sent from the Conners Creek Power Plant of the Detroit Edison Co., Detroit, Mich.

Sample 2 was collected by electrical precipitators. The coal is also from Districts 8 and 3 in Ohio and northern West Virginia and is processed through pulverizing mills so that 70 per cent passes the No. 200 sieve. The sample was sent from the Marysville Power Plant of the Detroit Edison Co.

Sample 3 was collected by multiple cyclone and electrical precipitators. The coal is also from Districts 8 and 3 in Ohio and northern West Virginia and is processed through pulverizing mills so that 70 per cent passes the No. 200 sieve. The sample was sent from the River Rough Power Plant of the Detroit Edison Co.

Sample 4 was collected by multiple cyclone and electrical precipitators. The coal is also from Districts 8 and 3 in Ohio and northern West Virginia and is processed through pulverizing mills so that 70 per cent passes a No. 200 sieve. The sample was sent from the St. Clair Power Plant of the Detroit Edison Co.

Sample 5 was collected by electrical precipitators. The coal is from District 8, east Kentucky and West Virginia and is processed through pulverizing mills so that 70 per cent passes a No. 200 sieve. The sample was sent from the Trenton Channel Power Plant of the Detroit Edison Co.

Sample 6 was collected by mechanical equipment. The coal is from western Kentucky and is crushed in bowl type crushers. The sample was sent from the Du Pont Neoprene Plant (adjoining Paddy's Run Station) south of Louisville, Ky.

Sample 7 was collected by mechanical precipitators. The coal is from northern Illinois and is burned in a Springfield boiler. This sample, from the Sixth Street Station in Cedar Rapids, Iowa, was sent by the Iowa Electric Light and Power Co.

Sample 8 was collected by mechanical equipment. The coal is from northern Illinois and is burned in a Babcock & Wilcox boiler. This sample, from the Sixth Street Station in Cedar Rapids, Iowa, was sent by the Iowa Electric Light and Power Co.

Sample 9 was collected by mechanical equipment. The fuel is a mixture of northern Illinois coal (65 per cent) and residue from furfural production (35 per cent) and is burned in a Babcock & Wilcox boiler. This sample, from the Sixth Street Station, Cedar Rapids, Iowa, was sent by the Iowa Electric Light and Power Co.

Sample 10 was collected by mechanical (centrifugal) precipitators. The coal is from Illinois and is pulverized in a ball mill prior to burning. The sample, from the Riverside

Station Power Plant at Davenport, Iowa, was sent by the Iowa-Illinois Gas and Electric Co.

Sample 11 was collected by mechanical precipitators (multicone dust collector). The coal is from Iowa (Monroe, Polk, Marion, and Mahaska Counties); it is unwashed steam coal which is pulverized and tangential fired. The sample was from the Des Moines Power Plant of the Iowa Power and Light Co.

Sample 12 was collected by mechanical equipment (VGR multicone). The coal (from southern Illinois) is washed, dried, and pulverized with Riley mills. The sample was sent from the Waterloo Power Plant by the Iowa Public Service Co.

Sample 13 was collected by mechanical precipitators. The coal is from Iowa and burned on spreader stokers without being pulverized. The sample was from Bridgeport Station Power Plant, Eddyville, Iowa, of the Iowa Southern Utilities Co.

Sample 14 was collected by mechanical precipitators (cyclone type). The coal is from several Missouri and Kansas mines. The coal is pulverized and burned in suspension in Combustion Engineering boilers. The sample was sent from the Hawthorne Station Power Plant of the Kansas City Power and Light Co., Kansas City, Mo.

Sample 15 was collected by mechanical precipitators (cyclone type). The coal is from Missouri and is pulverized and burned in suspension in Combustion Engineering boilers. The sample was from Montrose Station Power Plant of the Kansas City Power and Light Co.

Sample 16 was sent by the Kansas City Power and Light Co., Kansas City, Mo. No data are available on the source.

Sample 17 was collected by electrical precipitators. The coal is from western Kentucky and is crushed in bail mills. The sample, from the Cane Run Power Plant south of Louisville, Ky., was sent by the Louisville Gas and Electric Co.

Sample 18 was collected by electrical precipitators from a dry-bottom-type of boiler using unwashed coal from western Kentucky. The sample was from Paddy's Run Power Station at Louisville, Ky., of the Louisville Gas and Electric Co.

Sample 19 was collected by mechanical collectors. The coal is from the Birmingham, Ala., area and is crushed in bowl crushers. The sample was from the Barry Power Plant at Mobile, Ala.

Sample 20 was collected on electrical precipitators from a dry-bottom-type of boiler using southern Illinois coal. For economic reasons, the boilers are operated at 110 per cent of rated capacity, with a lowering of combustion efficiency. This sample was from the Venice Station Power Plant near St. Louis, Mo., of the Union Electric Co.

Sample 21 was collected by electrical precipitators. The coal is from southern Illinois and is crushed in a bowl crusher. The sample was from the Meramec Station Power Plant south of St. Louis, Mo. (near Jefferson Barracks), of the Union Electric Co.

Sample 22 was from the Oak Street Power Station by the Wisconsin Electric Power Co. No data are available on the source of coal or method of fly ash collection.

TABLE III.—CHEMICAL ANALYSIS, WEIGHT PER CENT.[a]

	Hydrated Limes	
	Dolomitic	Calcitic
Silicon dioxide	0.4	0.28
Iron and aluminum oxide	0.3	0.6
Magnesium oxide	31.8	0.59
Sulfur trioxide	1.1	0.25
Carbon dioxide	1.0	no data
Total calcium oxide	48.8	73.82
Available calcium oxide	47.1	70.3
Loss on ignition	17.1	24.1
Combined H_2O	18.0	no data

[a] Supplied by the U. S. Gypsum Co.

Limes:

The two commercial-grade limes used in this investigation were furnished by the U. S. Gypsum Co. One is hydrated calcitic lime, $Ca(OH)_2$, brand name Kemikal, and the other is type N monohydrate dolomitic lime, $Ca(OH)_2 + MgO$, brand name Kemidol. The calcitic lime was produced at New Braunfels, Tex., and the dolomitic lime was produced at Genoa, Ohio. The typical properties of these limes are shown in Table III. Commercial-type limes were used because the primary purpose of this investigation was to compare relative strengths obtained from mixtures with the different fly ashes, to facilitate selection of fly ashes for future field and laboratory tests of soil - lime - fly ash stabilization.

Water:

Distilled water was used so that results could be reproducible and so that there would be no possibility of the lime and fly ash reaction being affected by impurities that might be present in tap water.

LABORATORY INVESTIGATION

Analyses:

The chemical analyses of most of the fly ashes were performed by the Robert W. Hunt Co. The sieve analyses of fly ashes for determining the percentage passing the No. 100, No. 200, and No. 325 mesh sizes were performed on two separate samples of each fly ash. One hundred grams of fly ash were sieved through the No. 100 and No. 200 sieves to determine the per cent passing through each. After shaking 5 min with a mechanical shaker, the sieves were washed and the residue on each sieve was dried in an oven at 105 C. The procedure for determining the percentage passing the No. 325 sieve was the same except that only 50 g of fly ash were used.

Proportioning and Mixing:

Two mix proportions 1:4 and 1:9 by weight of lime and fly ash, were used with each fly ash for both types of lime. The moisture contents of the mixes were computed as percentages of the total dry weight of ingredients. Sufficient materials were added to make 4 test specimens of each mixture for preliminary work and 10 specimens for the final work. The materials to be tested were first dry mixed for 30 sec in a Hobart mixer (model C-100) at low speed and then hand scraped. The calculated volume of water was added and mixed for 2 min. This mix was hand scraped and then mixed for an additional 2 min.

Molding:

The strength test specimens were molded cylinders 2 ± 0.05 in. high and 2 in. in diameter. The wet lime and fly ash mixture was placed in the molding cylinder in one layer and compacted five times on each end by a 5-lb hammer dropping from a height of 12 in. (3,5). The double plunger action of the apparatus resulted in uniform density throughout the specimen. The resulting density was approximately equal to standard Proctor density (5). After molding, the specimen was extruded, weighed to the nearest 0.1 g, and then measured to the nearest 0.001 in. During molding, a wet cloth was kept over the bowl to prevent drying of the mixture.

Curing:

Preliminary Investigation.—Specimens to be moist cured were wrapped in waxed paper, sealed with cellophane tape, and placed in a humidity room in which the temperature was 70 ± 5 F and the humidity was above 80 per cent. Specimens were cured 7 days before being totally immersed in water for 1 day.

Specimens to be oven cured were wrapped in Saran wrap (or two layers of waxed paper), sealed with cellophane tape, and placed in an oven at 100 F for 6 days before 1-day immersion in water. Only a few mixtures were oven cured.

Final Investigation.—All specimens were wrapped in waxed paper, sealed with cellophane tape, and placed in a humidity room in which the temperature was 70 ± 5 F and the humidity was over 80 per cent. Specimens were allowed to cure for periods of 7 days, 28 days, or 4 months before 1-day immersion.

Testing:

All specimens were tested for unconfined compressive strength at a rate of load travel of 0.1 in. per min. This testing procedure was selected because of its simplicity. Three specimens were tested for each condition, and average strengths were recorded. This is in accordance with ASTM Method of Test C 109[6] which requires a minimum of 3 specimens for each set of curing conditions.[4] Specimens that differed by more than 10 per cent from the average value of test specimens made from the same mix and tested at the same age were not considered in determining compressive strength. If two specimens were rejected, new specimens were prepared.

Procedure:

Preliminary Investigation.—The work done during the preliminary study was to determine the moisture-strength and moisture-density relationships. Three specimens were molded at a given moisture content, weighed, and measured, then retained for strength tests after 7 days curing and 1 day immersion. This procedure was continued by increasing moisture content 4 to 5 per cent each time. Moisture-density and moisture-strength curves were then plotted to determine the optimum moisture contents for density and strength.

Final Investigation.—The specimens for final work were molded at the moisture contents found in the preliminary work. When the optimum moisture contents for strength and density did not coincide, a compromise was made by selecting a moisture content which minimized the total percentage deviation from both maximum strength and maximum density. An example of this procedure is given in Appendix I. Nine specimens of each mix were molded at the selected moisture content, wrapped, and placed in the humidity room. Specimens were cured as follows, before 1-day immersion and strength testing: specimens Nos. 1, 4, and 7, 7 days; specimens Nos. 2, 5, and 8, 28 days; and specimens Nos. 3, 6, and 9, 4 months.

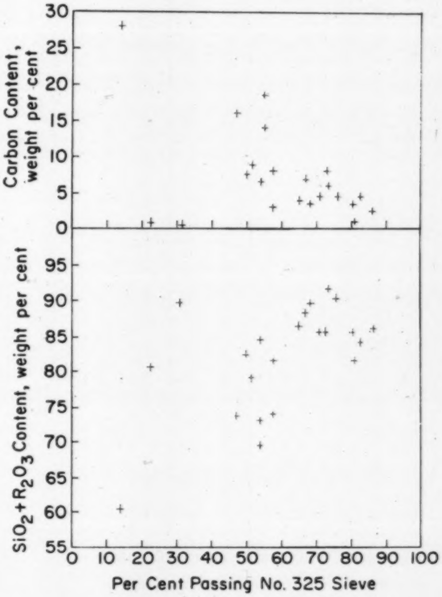

FIG. 1.—Comparison Between Fineness and Chemical Composition of Fly Ashes Used.

PRELIMINARY INVESTIGATION

Fly Ash Analyses:

The results of physical and chemical analyses of the fly ashes were compared to determine if there was any good correlation between physical properties and chemical composition. The per cent carbon and the per cent $SiO_2 + R_2O_3$

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[6] Method of Test for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. Cube Specimens) (C 109 – 58), 1958 Book of ASTM Standards, Part 4, p. 124.

($R_2O_3 = Al_2O_3 + Fe_2O_3$) in each fly ash were compared with the per cent of the total sample passing the No. 200 and No. 325 sieves (Fig. 1). The sieve analyses give indications of both the per cent carbon and the per cent reactive materials ($SiO_2 + R_2O_3$).

TABLE IV.—COMPARISON OF PHYSICAL PROPERTIES AND CHEMICAL COMPOSITION OF FLY ASHES ACCORDING TO METHOD OF COLLECTION.[a]

Property	Electrical	Mechanical	Electrical-Mechanical
Passing No. 100 sieve	96.45 (91.6–99.4)	90.11 (53.0–98.2)	98.1 (97.2–99.0)
Passing No. 200 sieve	87.35 (73.7–94.5)	76.10 (24.7–91.6)	93.73 (93.2–94.1)
Passing No. 325 sieve	73.3 (47.0–86.1)	49.85 (14.0–80.2)	72.5 (68.2–75.7)
Specific gravity	2.39 (2.26–2.60)	2.48 (2.04–3.43)	2.54 (2.39–2.61)
Specific surface	3628 (2539–4550)	2442 (576–4550)	2913 (2720–3070)
SiO_2, per cent	45.54 (35.94–57.50)	35.74 (11.26–43.72)	43.5 (41.6–47.0)
Fe_2O_3, per cent	17.56 (6.0–22.30)	28.20 (11.9–68.4)	23.10 (18.40–25.84)
Al_2O_3, per cent	19.68 (10.0–23.4)	15.58 (0.90–34.7)	23.79 (22.48–24.90)
CaO, per cent	4.87 (1.2–6.9)	6.54 (2.1–12.27)	2.18 (1.65–2.68)
MgO, per cent	1.00 (0.80–1.6)	0.75 (0.17–1.32)	1.30 (0.84–2.08)
SO_3, per cent	1.60 (0.45–2.3)	1.79 (0.8–3.4)	0.68 (0.55–0.79)
C, per cent	6.07 (1.0–15.59)	8.56 (0.15–28.06)	4.67 (3.5–6.0)

[a] Upper figures are averages. Lower figures indicate maximum and minimum.

The various fly ash collection methods were compared to determine the influence of this variable on physical properties and chemical composition of the fly ashes used. Table IV shows the comparison of electrical, mechanical, and combination electrical-mechanical collection methods. Generally the fly ashes collected by electrical methods are finer and hence contain more reactive material and less carbon than the fly ashes collected by mechanical methods. The three fly ashes collected by combination electrical-mechanical methods appear to have physical properties and chemical properties similar to the fly ashes collected by electrical methods. However, with only three fly ashes collected by electrical-mechanical methods available, no definite conclusions should be drawn.

The specific surface of each fly ash sample was compared with the per cent carbon and the per cent passing No. 325 sieve as shown in Fig. 2, but the correlation was not good. The fly ashes that varied considerably from the others were those that were exceptionally coarse (samples 10 and 13) or had exceptionally high carbon content (samples 7 and 13). It appears that specific surface is a function of fineness and carbon content. It is believed that the specific surface is influenced by both the external and internal surfaces of particles of carbon (2). Consequently, increase in the carbon content of fly ash may be accompanied by an increase in total surface area, even though the external surface area and the actual fineness of the fly ash decrease

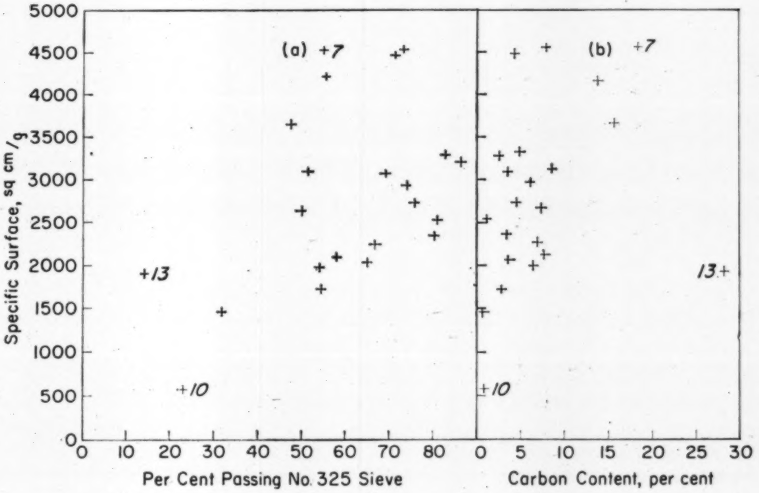


FIG. 2.—Comparison of Specific Surface with Carbon Content and Fineness of Fly Ashes Used.

TABLE V.—MOLE FRACTIONS OF CHEMICAL COMPOSITION OF FLY ASHES USED.[a]

Fly Ash	Chemical Components[a]						
	SiO_2 (60)[c]	Fe_2O_3 (160)[a]	Al_2O_3 (102)[c]	CaO (56)[c]	MgO (40)[c]	SO_3 (80)[c]	C (12)[c]
No. 1	40.9	9.3	14.4	2.3	3.1	0.5	29.5
No. 2	38.0	7.9	12.6	2.2	1.2	0.7	37.4
No. 3	52.9	7.8	15.8	2.0	1.4	0.5	19.6
No. 4	45.5	10.6	14.3	3.1	1.6	0.6	24.3
No. 5	58.6	2.3	13.3	1.3	1.2	0.4	22.9
No. 6	47.4	9.2	13.8	6.6	1.2	2.9	18.9
No. 7	23.3	4.0	6.0	5.7	0.9	0.7	59.4
No. 8	36.2	9.1	12.4	3.7	1.5	1.5	35.6
No. 9	38.5	6.1	8.7	5.7	1.7	0.7	38.6
No. 10	19.7	45.4	0.9	23.1	0.6	4.2	6.1
No. 11	56.6	19.5	10.9	8.8	0.7	2.5	1.0
No. 12	29.8	4.7	8.2	2.7	0.2	0.6	53.8
No. 13	14.6	4.3	3.0	4.6	0.5	0.4	72.6
No. 14	42.6	19.7	5.5	6.9	1.6	1.2	22.5
No. 15	45.1	13.1	8.1	14.4	1.3	1.6	16.4
No. 16	37.3	7.3	6.8	10.5	0.4	1.4	36.3
No. 17	46.1	6.5	12.7	7.5	1.7	1.8	23.7
No. 18	49.6	8.8	16.0	7.1	1.4	2.0	15.1
No. 19	39.3	4.2	19.3	2.2	1.9	0.6	32.5
No. 20	25.4	5.2	7.5	5.2	0.9	1.0	54.8
No. 21	63.9	9.5	7.3	8.5	3.0	1.6	6.2
No. 21	42.4	7.9	11.3	5.7	1.0	0.6	31.1

[a] Based on determined composition only.
[b] By per cent of assumed total moles present.
[c] Formula weights based on molecular weights of elements in each components (**13**).

(2). Therefore, specific surface was not used in further correlation studies.

Since the correlation studies also were to be made on the basis of chemical composition of fly ashes used, a table of mole fractions was prepared (13). For this study, the weight per cent of unknown chemical components (approximately 5 per cent) was neglected. The weight per cent of each component was divided by the formula weight (Table V) to obtain the mole fraction. The mole fractions of chemical components are also shown in Table V.

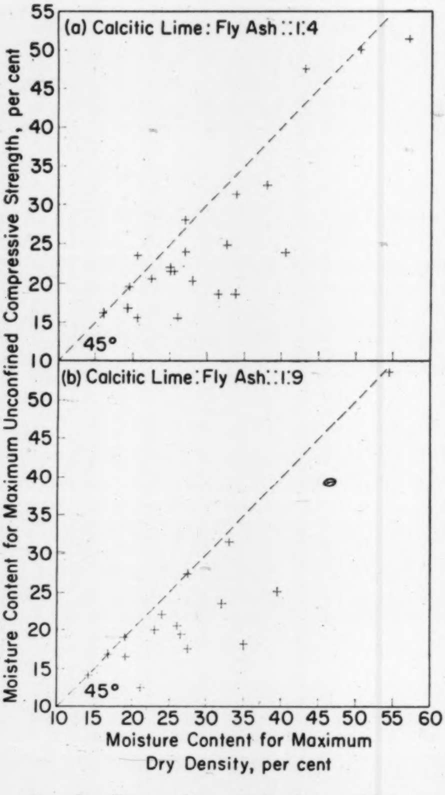


FIG. 3.—Comparison of Optimum Moisture Contents for Maximum Strength and Maximum Dry Density for Calcitic Lime - Fly Ash Mixtures.

Moisture-Density and Moisture-Strength Studies:

Initially, moisture-density and moisture-strength studies were made for all fly ashes with calcitic hydrated lime at ratios of 1:4 and 1:9. Mixtures for which all specimens failed during immersion after moist curing for 7 days at 70 F were reevaluated after 6 days at 100 F.

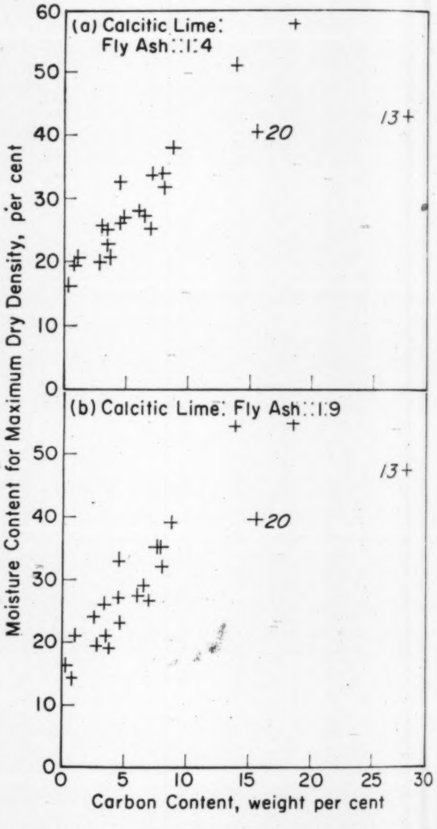


FIG. 4.—Carbon Content of Fly Ashes Versus Optimum Moisture Content for Maximum Dry Density of Calcitic Lime - Fly Ash Mixtures.

Five representative fly ashes, samples 2, 6, 7, 11, and 19, were selected for studies with dolomitic lime in a 1:4 ratio. Since the optimum moisture contents for dolomitic lime - fly ash mixtures and calcitic lime - fly ash mixtures are approximately the same, also verified by other investigators (14), it was decided to use the same moisture contents for both limes.

It was observed that the moisture content for maximum strength was generally below the moisture content for maximum dry density. This relationship is shown graphically in Fig. 3. It should be pointed out that in one case, sample 9, the moisture content for maximum dry density was not obtained because the specimens were too plastic and slumped slightly, making measurements inaccurate.

The moisture content for maximum dry density of lime - fly ash mixtures correlates reasonably well with the per cent carbon content (see Fig. 4), and to a lesser extent with specific surface of the fly ashes. This relationship between moisture content and carbon content agrees with previous findings (2,4). Some of the exceptions to this are samples 13 and 20, which were extremely coarse. These observations seem to indicate that the molding moisture requirement for lime - fly ash is a function of the carbon content and the fineness of the fly ash.

It was observed in molding the lime - fly ash mixtures that in some cases the mix behaved as a poorly graded sand and in other cases "bulked" (for example, sample 7). This "bulking" was not unexpected, since many of the fly ash particles are spherical in shape. The moisture-density curves generally were very flat over a range of 5 to 8 per cent of moisture content, with a gradual slope on the dry side and a steep slope on the wet side of optimum moisture content.

FINAL INVESTIGATION

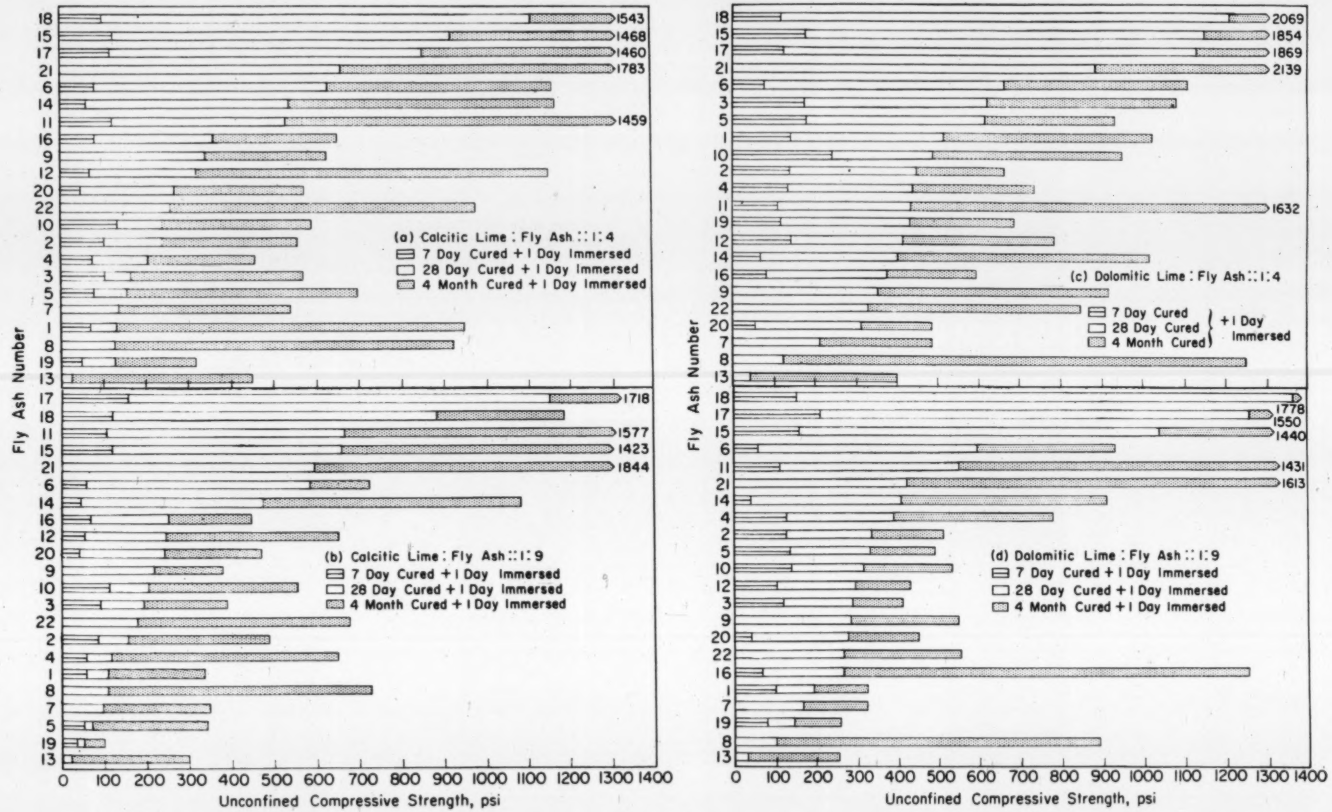
The fly ashes were mixed with both calcitic and dolomitic limes in ratios of 1:4 and 1:9 by weight, at moisture contents previously determined, and specimens were prepared for unconfined compression testing. The results of the unconfined compression tests were analyzed to determine the relative strengths with different fly ashes and the influence on strength of fly ash physical properties and chemical composition and type or quantity of lime.

Unconfined Compression Test Results:

The strengths after immersion for calcitic lime - fly ash mixtures are shown in Fig. 5 (*a*) and (*b*) and for dolomitic lime - fly ash mixtures in Fig. 5 (*c*) and (*d*), arranged in order of decreasing 28-day cured strengths. It should be kept in mind that the strengths are for lime - fly ash mixtures with no soil; the presence of soil could greatly influence the strengths.

The 7-day strength results for calcitic lime - fly ash mixtures with fly ash samples 1 through 5 were much lower than those obtained during the preliminary investigation, probably a result of low temperatures (averaging 65 to 67 F) during the first week of curing the final investigation specimens. It is believed that temperature variations did not seriously influence strengths of calcitic lime - fly ash mixtures with samples 6 through 22 or any of the dolomitic lime - fly ash mixtures. The results of strength tests for samples 1 through 5 mixed with calcitic lime will not be included in any calculations for this study because of their doubtful validity.

A comparison of unconfined compressive strengths in Fig. 5 (*a*) to (*d*) indicates that there is no correlation between 7-, 28-, and 120-day strengths. This agrees with previous findings (8). Some fly ashes react quickly with lime and achieve maximum strength early, whereas other fly ashes react slowly and attain near maximum strengths after



(*a*) Calcitic lime - fly ash ratio 1:4.
(*b*) Calcitic lime - fly ash ratio 1:9.

(*c*) Dolomitic lime - fly ash ratio 1:4.
(*d*) Dolomitic lime - fly ash ratio 1:9.

FIG. 5.—Unconfined Compressive Strength of Specimens of Lime - Fly Ash Mixtures Moist Cured 7 Days, 28 Days, and 4 Months and Immersed in Water 1 Day.

long curing times. The speed of the reaction would be greatly influenced by the solubility of the chemical substances present at the start of the reaction (**9**).

The 7-day strengths were not used in correlation studies because of the temperature variations that occurred during the final investigation and because in such a short period the pozzolanic activity has not developed sufficiently. In the presentation of results more emphasis is given to those obtained after 28-day curing, the standard curing period used with these materials.

TABLE VI.—COMPARISON OF STRENGTHS FROM IOWA-PRODUCED FLY ASHES WITH STRENGTHS FROM OTHER FLY ASHES.

Lime - Fly Ash Mixture	Average 28-Day Unconfined Compressive Strengths, psi[a]		
	Iowa Fly Ashes[b]	Other Mechanically Collected Fly Ashes	Other Fly Ashes
Calcitic lime - fly ash, 1:4	245	553	571[c]
Calcitic lime - fly ash, 1:9	222	444	507[c]
Dolomitic lime - fly ash, 1:4	295	662	635
Dolomitic lime - fly ash, 1:9	245	547	505

[a] After 1 day immersion.

[b] All mechanically collected.

[c] Does not include fly ashes 1 through 5.

The Iowa-produced fly ashes are compared with other fly ashes in Table VI. Since all the Iowa fly ashes were collected by mechanical methods, a comparison with other mechanically collected fly ashes is included.

In both comparisons, the Iowa-produced fly ashes are inferior pozzolans; the Iowa fly ashes, however, are a random selection, whereas most of the other fly ashes have been accepted for commercial use in concrete.

Effects of Fly Ash Physical Properties and Chemical Composition on Pozzolanic Behavior:

Comparisons were made between each fly ash's physical properties and chemical composition and the 28- and 120-day strengths obtained in lime - fly ash mixtures. Since the lime - fly ash reaction is chemical, it seems a logical assumption that the chemical composition and physical properties of a fly ash should correlate with strengths obtained with the lime-fly ash mixtures.

An attempt was made to divide the fly ashes into several groups having similar characteristics (for example, per cent passing No. 325 sieve, or silica content). It became obvious that the number of variables and range within each variable were too great for the number of observations. For this reason, all fly ashes were taken as a group for the correlation studies.

Values of 28-day cured strengths of lime - fly ash mixtures plotted against the weight per cent of fly ash passing the No. 325 sieve showed a definite relationship to strength. Strength plotted against values of per cent passing the No. 325 sieve divided by per cent retained on No. 325 sieve is shown in Figs. 6 and 7. There are two factors contributing to the good correlation: The first is that fineness has been found to be a general indicator of those chemical components of fly ash which are considered to be reactive; the second is that the finer particles increase the surface area and hence increase the rate of reaction of fly ash with lime. The same condition was found for the 4-month cured specimens.

Values of fly ash specific surface were not plotted against strengths because the preliminary studies indicated that specific surface was a function of fineness and carbon content. Previous studies of fly ash in cement - fly ash mortars indicated that no definite correlation existed between specific surface and unconfined compressive strength (2).

In molding specimens, it was observed that several fly ashes having approximately the same specific gravity gave widely differing densities in compacted lime - fly ash mixtures. Later, in testing and in attempting to correlate strengths with fly ash characteristics, it was found that some of the lime - fly ash mixtures which gave exceptionally high strengths also gave exceptionally high densities. Conversely some of those fly ashes which gave low strengths also gave low densities.

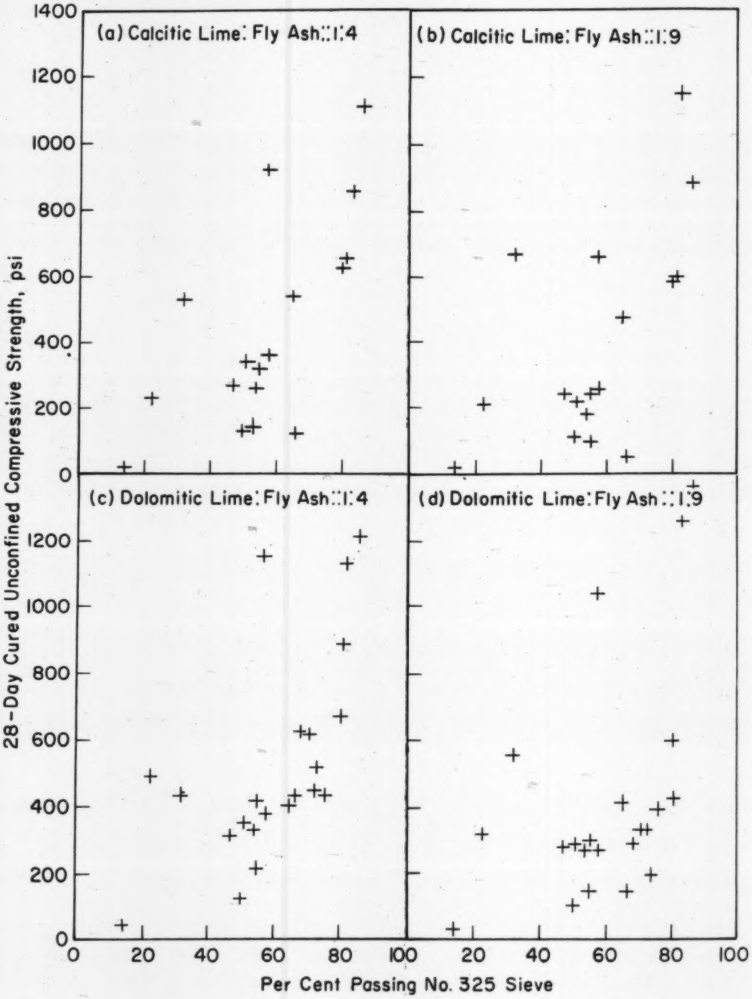


FIG. 6.—Comparison of 28-Day Unconfined Compressive Strength of Lime - Fly Ash Mixtures with Fineness of Fly Ashes.

The per cent solids and void ratios of compacted lime - fly ash mixtures were calculated from the dry densities of lime - fly ash mixtures at the time of molding and from the appropriate specific gravity of each lime - fly ash mixture. Closer analysis indicated that those fly ashes collected electrically generally gave mixes with a higher percentage of solids than fly ashes collected mechanically. Values of strengths were plotted against the product of per cent solids in the lime - fly ash mixtures and the weight per cent of fly ash passing the No. 325 sieve, and against the product of per cent solids in the lime - fly ash mixture and the weight per cent of fly ash passing the No. 325 sieve divided by the weight per cent of fly ash retained on the No. 325 sieve (Figs. 8 and 9). The authors assumed that strength of lime - fly ash mixes increases linearly with increase of per cent solids. Two explanations are offered for the variations in per cent solids: particle size distribution of the fly ash and electrostatic charges on the fly ash particles. If the lime - fly ash mixture is well graded (influenced by gradation of both lime and fly ash particles) a much denser mix (higher per cent solids) will be obtained than with a poorly graded mix. If there is an electrostatic charge on the fly ash particles,

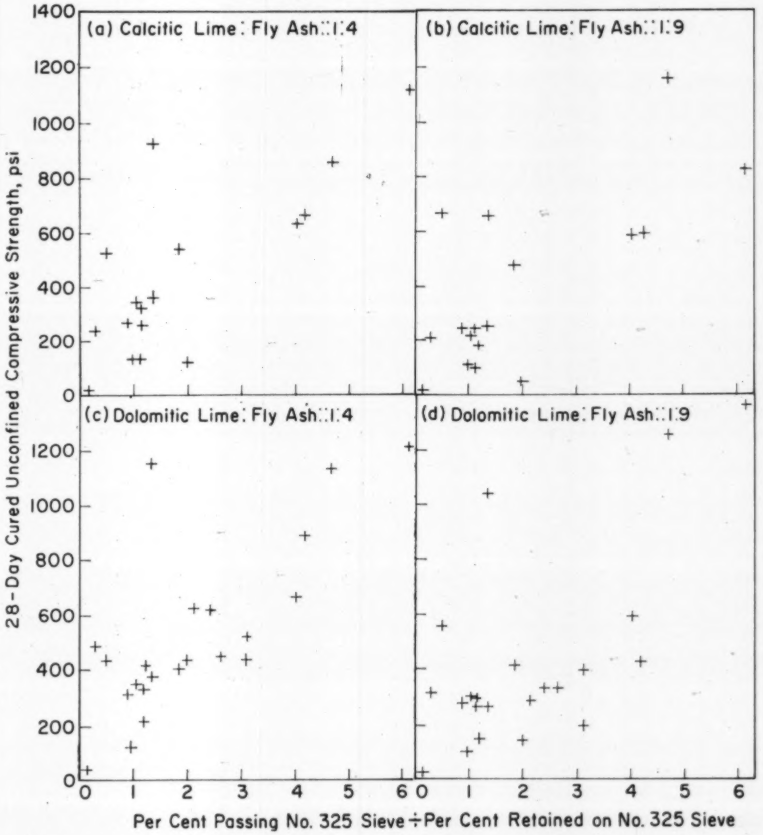


Fig. 7.—Comparison of 28-Day Unconfined Compressive Strength of Lime - Fly Ash Mixtures with Per Cent of Fly Ash Passing No. 325 Sieve Divided by Per Cent Retained on No. 325 Sieve.

there is a possibility that like charges on all particles could cause repulsion, thus preventing attainment of high per cent solids.

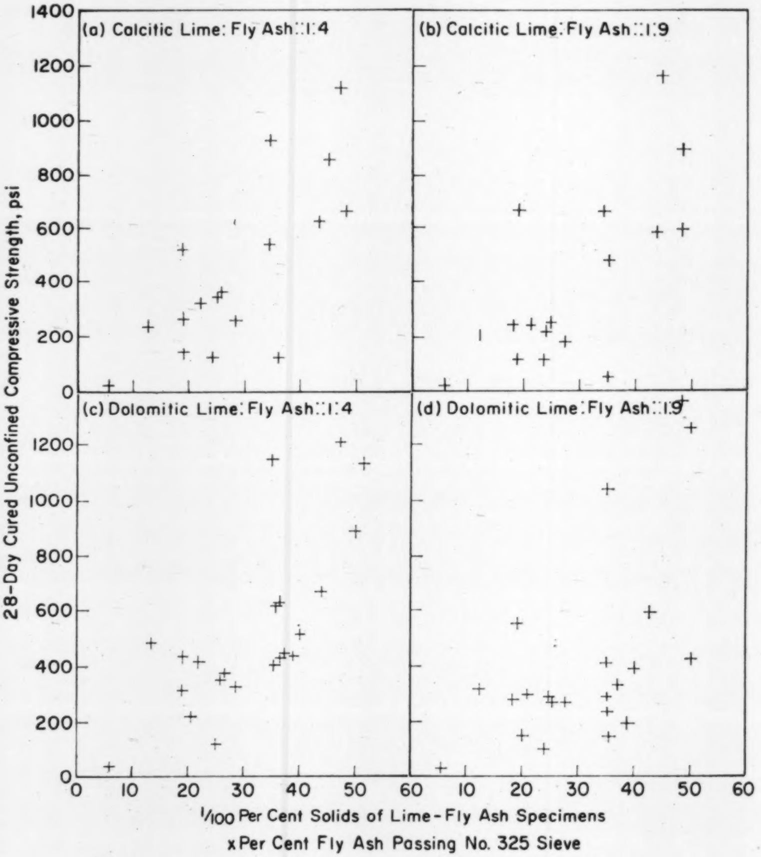


FIG. 8.—Comparison of 28-Day Unconfined Compressive Strength with Per Cent Solids of Lime - Fly Ash Mixtures Times Per Cent of Fly Ash Passing No. 325 Sieve.

The strengths were plotted against the total amounts of each of the inorganic components of the fly ashes given in Table II. The amounts of inorganic components were given both in weight per cent and in mole fraction. No definite relationship was found between strength of lime - fly ash mixtures and the weight per cent of the different fly ash components. However, the mole fraction of silica correlated fairly well with strength. Plots of silica content (weight per cent and mole fraction) are shown in Figs. 10 and 11. This gives an indication that mole fractions are more favorable for correlation studies.

Brink and Halstead (2) have shown that the strengths of fly ash - cement mortars (with standard sand) are adversely affected by fly ashes having high carbon contents. A similar trend is evident in Figs. 12 and 13, in which the relation between the 28-day pozzolanic strength and carbon content is shown. As with silica content, the plot of carbon content in mole fraction versus strength of lime - fly ash mixtures gives a slightly better relationship than carbon content in weight per cent versus strength. Three factors are thought to be responsible for the reduced strength with increased carbon content. First, the organic material in fly ash does not react with $Ca(OH)_2$ (8). Second, the organic material occurs predominantly in the coarse fractions of a fly ash and its presence indicates coarse inorganic material of low reactivity. Third, the carbon particles are structurally weak.

The triangular chart concept was applied in which the various chemical components were grouped into three categories according to assumed influence on strength: favorable, neutral and unfavorable. The triangular chart procedure was similar to that used for textural classification of soils in which the three sides of an equilateral triangle are graduated from 0 to 100 per cent representing sand, silt, or clay sizes. Triangular charts were prepared (see Appendix II for procedure) for weight

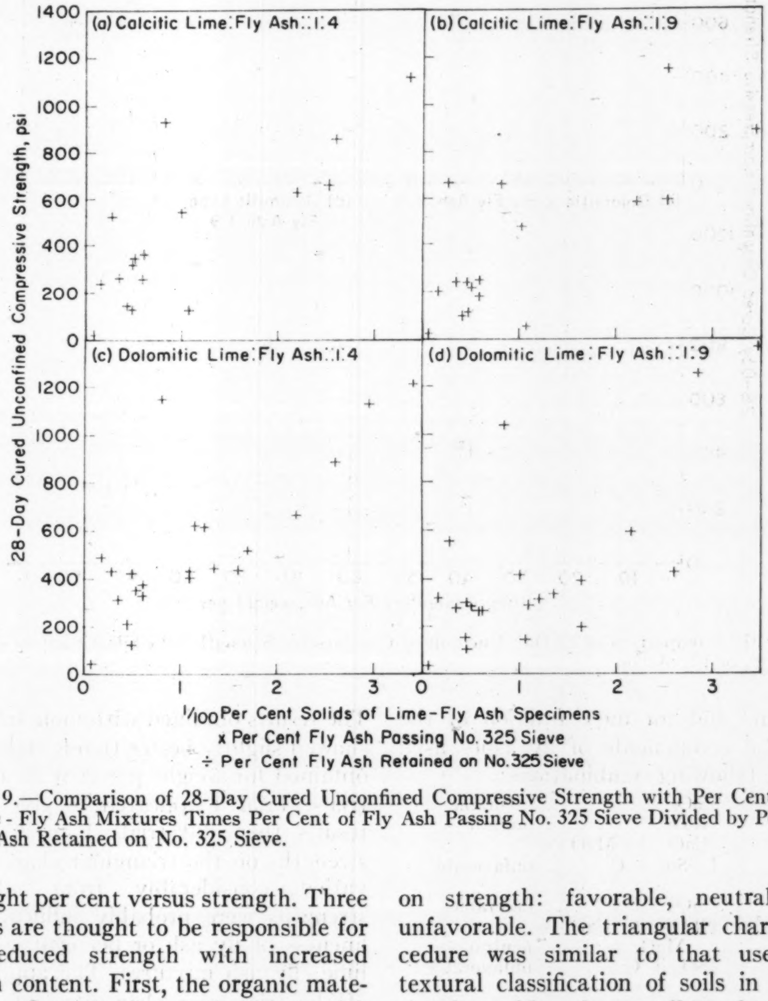


FIG. 9.—Comparison of 28-Day Cured Unconfined Compressive Strength with Per Cent Solids of Lime - Fly Ash Mixtures Times Per Cent of Fly Ash Passing No. 325 Sieve Divided by Per Cent of Fly Ash Retained on No. 325 Sieve.

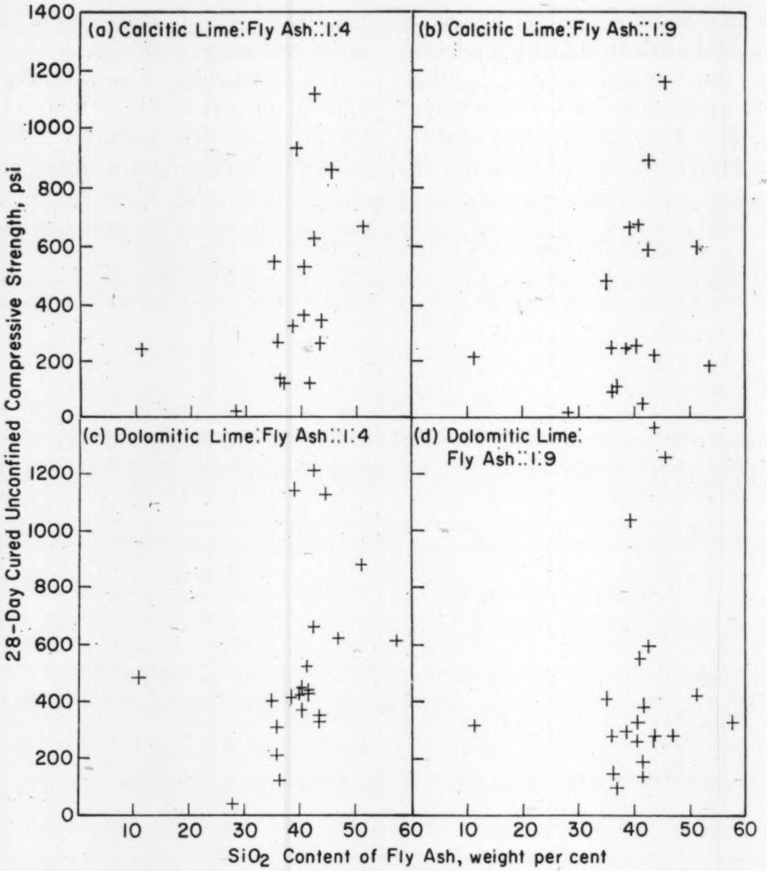


FIG. 10.—Comparison of 28-Day Unconfined Compressive Strength with Silica Content (Weight Per Cent) of Fly Ashes.

per cent and for mole fraction of the chemical components of fly ashes used for the following combinations:

Trial 1..... SiO_2 .... favorable
R_2O_3 .... neutral
$CaO + MgO +$
$So_3 + C$ .... unfavorable

Trial 2..... SiO_2 .... favorable
$R_2O_3 + CaO +$
MgO .... neutral
$SO_3 + C$ .... unfavorable

Trial 3..... $SiO_2 + Al_2O_3$ ..... favorable
$Fe_2O_3 + CaO +$
MgO .... neutral
$SO_3 + C$ .... unfavorable

Trial 4..... $SiO_2 + R_2O_3$ ...... favorable
$CaO + MgO$ ..... neutral
$SO_3 + C$ .... unfavorable

The results obtained with mole fractions showed slightly better trends than those obtained for weight per cent in all four trials. Trial 4 appeared to give better results than did trials 1 to 3. Those strengths on the triangular chart which varied considerably from adjacent strengths were probably influenced by fineness of fly ash or per cent solids of lime-fly ash mixtures. The application of the triangular chart concept to the correlation of chemical components of fly ashes with strength of lime-fly ash mixtures appears to be promising.

Twenty-eight-day strengths were also plotted against $SiO_2 + R_2O_3$ contents (weight per cent and mole fraction) of the fly ashes. The results using the mole fraction are shown in Fig. 14. As before, the mole fraction percentages gave better trends, showing a definite relationship between increase in strength and increase in $SiO_2 + R_2O_3$.

The following combinations were tried:

Per cent of fly ash passing No. 325 sieve divided by carbon content,

Product of per cent solids in the lime - fly ash mixture and per cent $SiO_2 + R_2O_3 - C$ in the fly ash, and

Product of void ratio of the lime - fly ash mixture and carbon content of the fly ash.

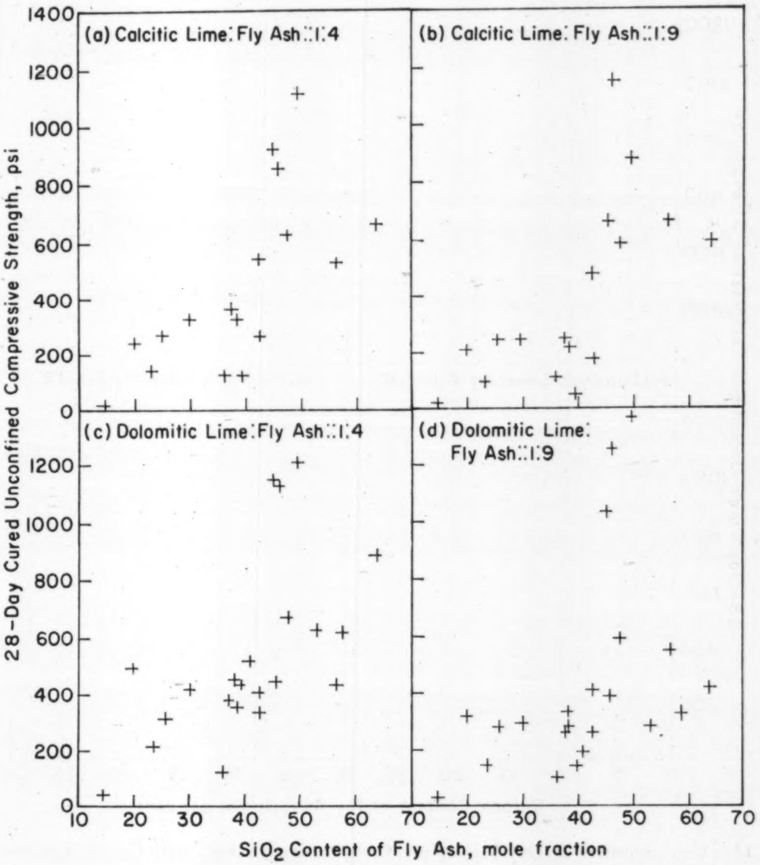


FIG. 11.—Comparison of 28-Day Unconfined Compressive Strength with Silica Content (Mole Fraction) of Fly Ashes.

The next correlations attempted were between unconfined compressive strength of lime - fly ash mixtures and combinations of the physical properties and chemical components of fly ashes which had shown the most promise in previous trials. None of these gave correlations that were considered to be better than those obtained in previous trials.

Variations of Type of Lime and Lime - Fly Ash Ratio.—The averages of the results indicate that the dolomitic lime gave 28-day strengths approximately 30 per cent greater than calcitic lime. This average figure is very misleading, because the use of dolomitic lime in lime-fly ash mixtures can give lower strengths than calcitic lime when mixed with some fly ashes.

compared with contents of SiO_2, CaO, MgO, and Ca:Mg ratio of fly ashes to determine if there was any correlation between chemical composition and effect of calcitic and dolomitic limes. Since no correlation between chemical composition of fly ash and effectiveness of the two limes was obtained, it is believed that one of the chemical components of fly ash not determined could have influenced the reaction. A good possibility is the alkali content (9).

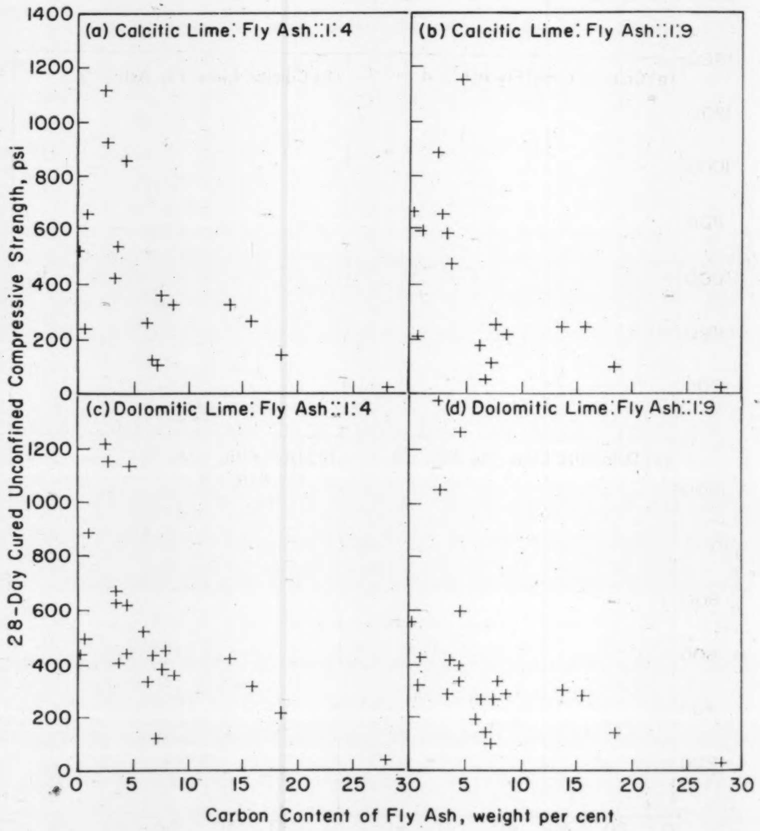


FIG. 12.—Comparison of 28-Day Unconfined Compressive Strength with Carbon Content (Weight Per Cent) of Fly Ashes.

The dolomitic lime-fly ash mixtures generally gave higher densities than the calcitic lime-fly ash mixtures. No correlation was found between the ratio of densities (dolomitic lime-fly ash to calcitic lime-fly ash) of compacted lime-fly ash mixtures and the ratio of strengths. The strength ratios were then

Tests were made to determine the alkalinity of the fly ashes using a Leeds

& Northrup pH meter and pH paper. No good correlations were obtained between pH and effectiveness of the limes. Variations in pH readings between the two test methods indicate the presence of electrostatic charges that could influence the lime-fly ash reaction. Further study should be made of this problem.

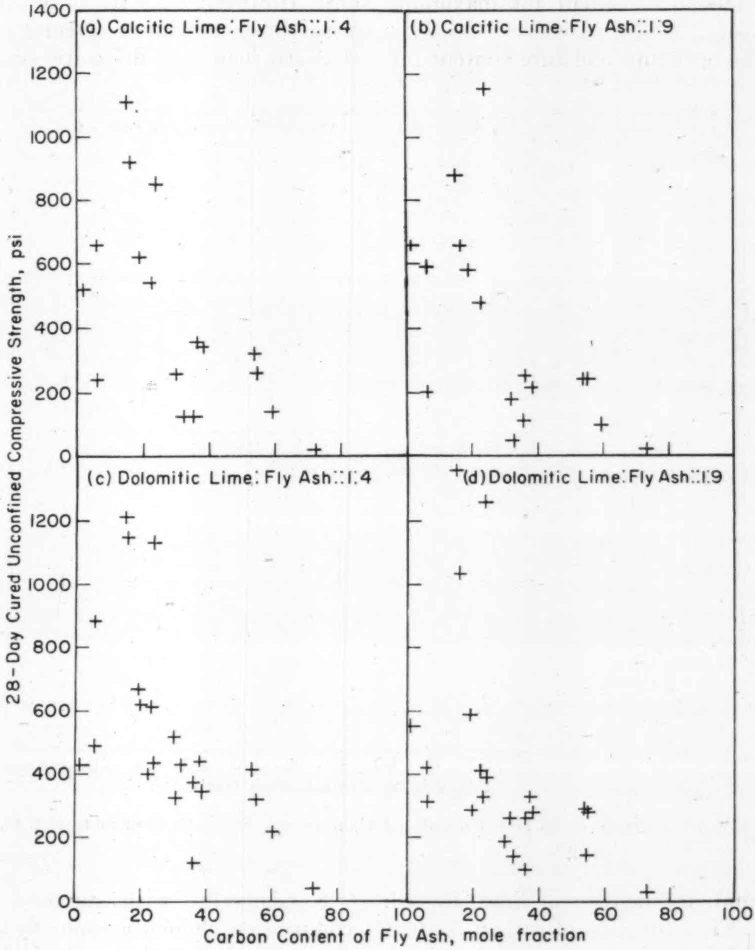


FIG. 13.—Comparison of 28-Day Unconfined Compressive Strength with Carbon Content (Mole Fraction) of Fly Ashes.

CONCLUSIONS

Results obtained from the investigations indicate the following:

1. Fly ashes collected in power plants by electrical methods generally are finer and contain less carbon than fly ashes collected by mechanical means.

2. The weight per cent of fly ash passing the No. 325 sieve gives a general

indication of the carbon content and amount of reactive material.

3. The optimum moisture content for maximum strength of lime-fly ash mixtures is generally slightly below optimum moisture content for maximum dry density.

4. The optimum moisture content for maximum dry density of lime-fly ash mixtures is a function of carbon content and fly ash fineness.

5. The average 28-day strengths obtained with seven Iowa-produced fly ashes are approximately 50 per cent of the corresponding strengths obtained with the other fly ashes tested.

6. No correlation was found between the chemical composition of fly ash and the influence on 28-day strength by varying the type of lime or the lime:fly ash ratio.

7. Generally, the use of dolomitic monohydrate lime produces strengths 30 per cent greater than calcitic hydrated lime. However, with some fly ashes calcitic lime produces greater 28-day strength than does dolomitic lime.

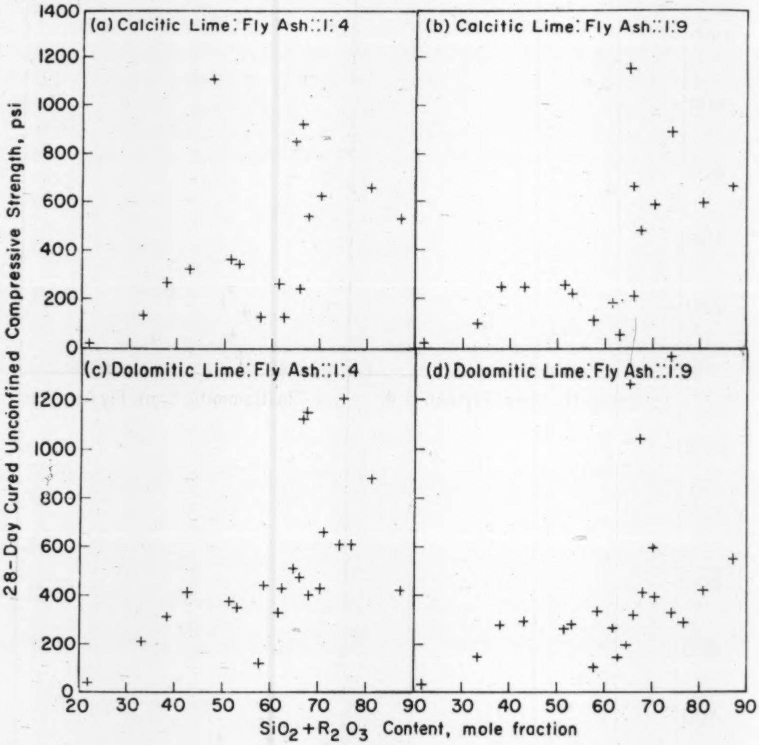


FIG. 14.—Comparison of 28-Day Unconfined Compressive Strength with $SiO_2 + R_2O_3$ Content (Mole Fraction) of Fly Ashes.

8. Generally, with standard Proctor compaction, dolomitic lime-fly ash mixtures are denser than calcitic lime-fly ash mixtures. No correlation was found between increased densities and strengths, comparing both calcitic and dolomitic limes.

9. No correlation was obtained between 7-, 28-, and 120-day strengths when lime-fly ash mixtures were cured at 70 F.

10. Compacted lime - fly ash mixtures generally have a higher percentage of solids with electrically collected fly ashes than with mechanically collected fly ashes.

11. The chemical composition of fly ash expressed in mole fractions correlates with strength better than does chemical composition expressed in weight per cent.

12. The application of the triangular chart concept to the correlation of chemical composition with 28-day strength appears to offer promising results.

13. Sieve analysis using No. 325 sieve, carbon content determined by loss on ignition, and compaction tests to determine per cent solids appear to be fairly reliable criteria of fly ash quality for soil lime - fly ash stabilization.

Acknowledgments:

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APPENDIX I

PROCEDURE FOR SELECTING MOISTURE CONTENTS FOR MOLDING STRENGTH SPECIMENS

The curves for unconfined compressive strength versus moisture content and dry density versus moisture content were plotted and optimum moisture contents for maximum strength and maximum dry density were determined. The moisture content for molding strength specimens is a compromise, and is the moisture content at which the total per cent deviation from maximum strength and maximum dry density is a minimum.

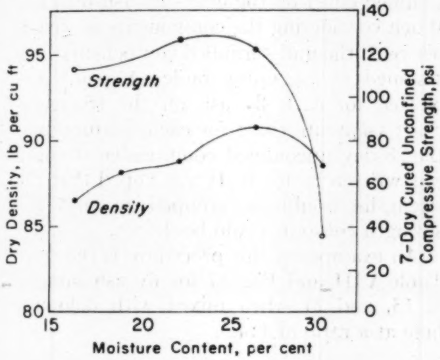

FIG. 15.—Molding Moisture Content Versus 7-Day Unconfined Compressive Strength and Dry Density for Fly Ash Sample 18 at Calcitic Lime to Fly Ash Ratio of 1:4.

TABLE VII.—TOTAL PER CENT DEVIATION FROM MAXIMUM STRENGTH AND MAXIMUM DRY DENSITY FOR FLY ASH SAMPLE 18 FOR CALCITIC LIME: FLY ASH RATIO = 1:4.

Moisture Content, per cent	Per Cent Deviation from Maximum Strength	Per Cent Deviation from Maximum Dry Density	Total Per Cent Deviation
22	3.05	2.53	5.58
23	0.76	2.09	2.85
24	0.00	1.43	1.43
25	0.76	0.77	1.53
26	2.29	0.33	2.62
27	6.1	0.00	6.1
28	15.3	0.33	15.63

An example of this procedure is shown for fly ash sample 18 in a calcitic lime - fly ash mix having a ratio of 1:4. The results of the moisture - strength and moisture - density study have been plotted on Fig. 15. The optimum moisture contents obtained are:

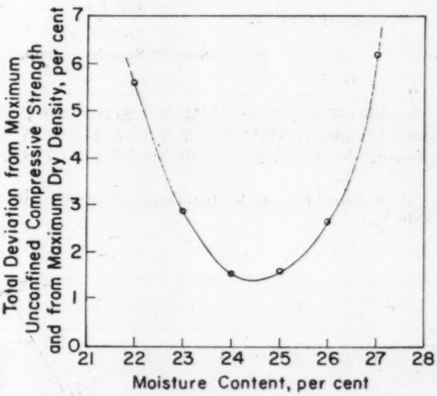

FIG. 16.—Molding Moisture Content Versus Total Per Cent Deviation from Both Maximum 7-Day Unconfined Compressive Strength and Dry Density for Fly Ash Sample 18 at Calcitic Lime to Fly Ash Ratio of 1:4.

Maximum confined compressive strength = 131 psi at 24 per cent moisture content

Maximum dry density = 91 lb per cu ft at 27 per cent moisture content

The total per cent deviations, calculated from Fig. 15, are shown in Table VII. The curve of total per cent deviation versus moisture content is shown in Fig. 16, which indicated a minimum point at a moisture content of 24.5 per cent.

APPENDIX II

USE OF TRIANGULAR CHART TO CORRELATE CHEMICAL COMPOSITION OF FLY ASHES WITH STRENGTH OF LIME - FLY ASH MIXTURES

TABLE VIII.—CHEMICAL COMPOSITION OF SELECTED FLY ASHES ACCORDING TO COMPONENT'S ASSUMED EFFECT ON LIME-FLY ASH REACTION.

Component Groups	Total Per Cent[a] of Component in Each Fly Ash		
	Sample 9	Sample 13	Sample 21
Favorable ($SiO_2 + R_2O_3$)	53.3	21.9	80.7
Neutral ($CaO + MgO$)	7.4	5.1	11.5
Unfavorable ($SO_3 + C$)	39.3	73.0	7.8

[a] Per cent by mole fraction as shown in Table V.

The chemical components of the fly ashes were divided into three groups: favorable, neutral, and unfavorable, according to an assumed effect on the lime - fly ash reaction. When considering the components in weight per cent, the undetermined components were assumed to be unfavorable. A point was plotted for each fly ash on the triangular chart (separate chart for each mixture) and the 28-day unconfined compressive strength was written beside it. It was hoped that the strengths would be grouped so that *iso*-strength contours could be drawn.

An example of this procedure is shown in Table VIII and Fig. 17 for fly ash samples 9, 13, and 21 when mixed with dolomitic lime at a ratio of 1:4.

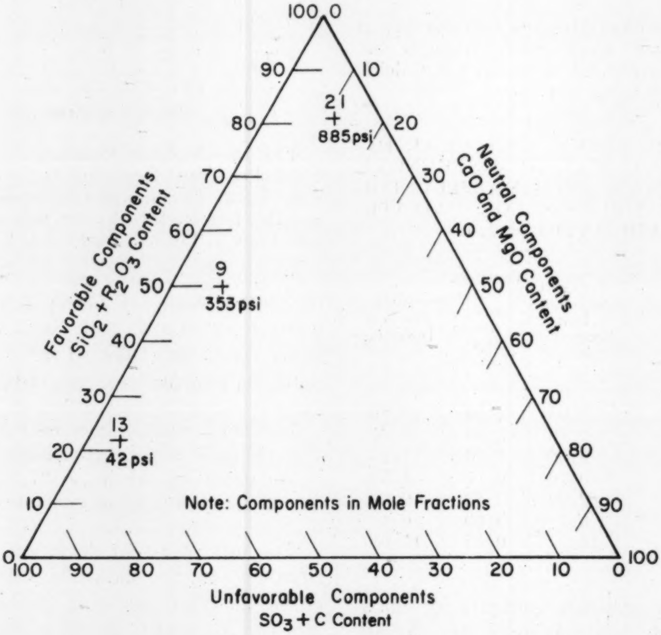

FIG. 17.—Comparison of Chemical Components with 28-Day Strength of Selected Dolomitic Lime - Fly Ash Mixtures (Ratio 1:4) by Use of Triangular Chart.

TESTING UNIFORMITY OF LARGE BATCHES OF CONCRETE*

By D. L. Bloem,[1] R. D. Gaynor,[1] and J. R. Wilson[1]

Synopsis

Multiple samples were secured from 51 full-sized batches of ready-mixed concrete. These were subjected to a variety of tests to measure differences in properties of the concrete at various points within each batch. Measurements included slump, unit weight, air content, coarse aggregate content, cement by centrifuge separation, water content by drying, and compressive strength. Supplementary measurements and calculations provided data on cement factor, sand-aggregate ratio, mixing water content, and water-cement ratio.

The regimen of sampling and testing was varied to determine which combinations provided practical means of evaluating concrete uniformity. The number of samples from a given batch ranged from two to twelve. For some samples, tests were made in duplicate or triplicate to assist in estimating the number needed to assure reliable comparisons. In some cases, all practicable operations of sampling and testing were conducted in the field. In others, a minimum of the work was done in the field, with samples being transferred to the laboratory for processing.

The data are presented in tabular form, and graphical comparisons are made among the measures of within-batch variation. The various tests are discussed from the standpoint of their utility in evaluating concrete uniformity. Recommendations are made for procedures to establish whether or not various portions of a given batch differ significantly in their properties.

Uniformity of concrete is essential if it is to achieve reliably the properties anticipated. For a batch to contain the correct amount of each ingredient is of little value if those ingredients are not uniformly blended to produce the correct proportions throughout.

Much attention is being directed toward devising ways of measuring uniformity. Subcommittee III-i on Ready-Mixed Concrete of ASTM Committee C-9 on Concrete and Concrete Aggregates has under development a set of criteria for within-batch uniformity of ready-mixed concrete produced in central mixers. It is intended to provide information on the amount of mixing required to produce adequate homogeneity. Use of this uniformity evaluation as an alternate to fixed time limits on mixing is being considered as a tentative revision of the ASTM Specifications for Ready-Mixed Concrete (C 94).[2]

In addition to establishing required mixing time for central mixers, uniformity tests may be used to indicate blade wear, study mixing speed, or establish efficient batching sequences.

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[2] ASTM Specifications for Ready-Mixed Concrete (C 94 – 58), 1958 Book of ASTM Standards, Part 4, p. 600.

Application might be extended to factors involved in handling and placing concrete, where segregation could undo the accomplishments of proper batching and adequate mixing.

Obviously, the need for uniformity tests extends to truck-mixed as well as central-mixed concrete. Both the producer and user have a stake in balancing economy against performance by using efficient mixing times, rates, and capacities. The Standards Committee of the National Ready Mixed Concrete Assn. recognized the importance of this problem about two years ago in the establishment of a Subcommittee on Mixer Efficiency. Its objective was to develop procedures for measuring within-batch variation of concrete and, if possible, to establish limits for adequate uniformity. Review of the literature revealed much valuable work in this connection but suggested that an organized program was needed to establish methods and criteria specifically for truck mixers.

In 1921, Hatt **(1)**[3] conducted tests on concrete mixers, investigating effects of time of mixing and variations in water content and speed of drum rotation on the consistency and strength of concrete.

In 1929, Dunagan **(2)** proposed a method for measuring the composition of fresh concrete. Cement, water, sand, and coarse aggregate are determined by a series of wash-separations and weighings in air and in water. The method is not widely used because of inaccuracies in determining immersed weights and in correcting cement content for the presence of fine sand.

Slater **(3)**, in 1931, made a study of concrete mixed at a central plant and transported in a truck conveyor for different periods. Samples were secured and tested by the Dunagan method. Measurements were made of the decrease in slump, apparent increase in cement content, and increase in strength of concrete during transportation.

Hollister **(4)**, in 1932, conducted tests on concrete mixed in truck mixers at different rates of rotation. The concrete was tested by the Dunagan method. It was observed that the relation of strength to water-cement ratio was the same at the different mixing rates. When water was added during mixing, strength was reduced in accordance with the water-cement ratio law.

A study of the effects of time of haul on strength and consistency of ready-mixed concrete was reported by Cook **(5)** in 1943. Concrete was mixed in a stationary mixer and hauled for various periods, up to 7 hr, in a truck agitator. Strength and slump tests were made at intervals, and concrete at the start and end of haul was analyzed by the Dunagan method. The effects of retempering concrete to restore slump were also studied. Contrary to Hollister's findings, strength was decreased less by the addition of retempering water than was expected from the increase in water-cement ratio. When water was not added, prolonged agitation reduced slump and increased strength.

A more recent investigation of truck mixers was reported by Walker and Bloem **(6)** in 1954. The effects on concrete uniformity of type of loading, size of load, number of revolutions, and speed of rotation were evaluated. The data supported the recommendation that mixer capacity ratings could be increased above the then accepted levels. Concrete uniformity was evaluated in terms of within-batch variations in slump, strength, density, air content, and coarse aggregate content.

In 1958, Timms **(7)** conducted a comprehensive study of truck mixer performance using methods similar to those of Walker and Bloem. Primary variables were rate and amount of mixing and size of batch. Sequence of loading and effects of slump on mixing efficiency were also studied.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

Additional very recent research in this field has been reported since initiation of the investigation which is the subject of this paper. Maxon (8) and Narrow (9) presented data on tests of uniformity of concrete from stationary mixers. Those researches involved within-batch comparisons of such characteristics as slump, air content, unit weight, strength, and cement content by centrifuge separation (10). A study of paving mixers, involving measures of within-batch uniformity by several highway departments, was recently described in a paper by Woolf (11).

While these researches fulfilled their objectives and contributed worthwhile information over the years, it was felt that additional study was needed, aimed specifically to setting up test methods and establishing limits for variations in truck-mixed concrete. Last year, the Research Committee of the National Sand and Gravel Assn. and National Ready Mixed Concrete Assn. (NRMCA) approved such a program for conduct by the Associations' Joint Research Laboratory at the University of Maryland. The Truck Mixer Manufacturers Bureau, through its representatives on the NRMCA Standards Committee, became interested in the study and agreed to underwrite a substantial share of its cost. This paper reports that research.

SCOPE OF RESEARCH

A crew of three men was specifically engaged and trained for this project. They were under the immediate direction of Milton H. Wills, Graduate Fellow of the Associations at the University of Maryland; and were assisted in the laboratory by the regular staff. The entire operation was under the supervision of coauthor R. D. Gaynor. Responsibility for organization of the project was in the hands of Stanton Walker, Director of Engineering of the two associations.

A total of 51 full-sized batches of ready-mixed concrete were sampled and tested. Local area members of the NRMCA cooperated by permitting the testing crew to watch all processes of batching and mixing and by arranging with contractors to allow sampling. All batches were from regular production, and no special controls were exercised over any phase of the operation. However, to the extent practicable, records were kept of pertinent information, such as batching sequence, time required for transportation, mixing and discharge, any additions of water during discharge, and relevant data on type of equipment, size of batch, and nominal characteristics of the concrete.

A truck was provided to facilitate accompaniment and observation of the concrete throughout its production, transportation, and discharge. Special covered racks were installed to permit the moving of samples and specimens to the laboratory for testing. A variety of tests were performed on each batch, with procedures varied to assist in evaluating their ability to discern differences within batches. Altogether, the study involved: approximately 300 slump tests, 370 compression tests of 6 by 12-in. cylinders, 125 unit weight measurements, 500 wash tests for aggregate content, 125 pressure meter air content determinations, 225 drying tests for water content, and 225 centrifuge tests for cement content. Calculations were made of other batch characteristics such as sand content, air-free unit weight of mortar, and water-cement ratio.

TESTING PROCEDURES

In most cases, either two or three samples were secured from each batch. Each sample consisted of about 175 lb of concrete collected in four metal buckets by intercepting the entire flow from the discharge chute. One bucket was used for the slump test,[4] made in duplicate. This concrete was then discarded. The remaining three buckets were covered with sheet plastic to prevent evaporation until the concrete was used for other tests. In general, these samples were moved immediately to the laboratory for processing but, in some cases, as much as possible of the testing was done in the field.

The three buckets of concrete representing each portion of the batch were transferred to a metal pan and recombined by shovel. A unit weight test was made in the $\frac{1}{4}$-cu ft base of a pressure air meter,[5] after which the same sample was tested for air content.[6] Finally, this $\frac{1}{4}$-cu ft of concrete was washed over a reinforced No. 8 sieve and the retained coarse aggregate was oven dried. The next day, the dried material was screened over a No. 4 sieve to permit calculating the percentage of coarse aggregate in the concrete. This procedure was similar to that used by several earlier investigators and was not unlike that employed by the California Division of Highways for checking coarse aggregate content of concrete.[7] From the data on this portion of the sample, the air-free unit weight of mortar was later calculated. Comparisons of that characteristic are used by the Bureau of Reclamation to evaluate performance of stationary mixers.[8]

A small portion (about 4 to $4\frac{1}{2}$ lb) of the concrete in the metal pan, representing a given location in the batch, was secured for determination of cement content by the centrifuge method. This test has been described[2, 9] and its significance discussed in detail in a recent paper in *Materials Research & Standards* (12). The test employs a heavy liquid, denser than the sand but less dense than the cement, to separate those two ingredients from a small amount of mortar extracted from the concrete. It provides a basis for within-batch comparisons of cement content, as used here; however, in its present state of development, it is not a suitable measure of cement factor level for purposes of checking conformance with specifications.

Another small portion of the concrete sample (about 3 to 4 lb) was subjected to drying to measure its water content. Its hardening was delayed by the addition of sugar and it was dried overnight at moderately high temperature, about 550 F. The dried material, after being weighed to determine the amount of water in the small sample, was later subjected to complete sieve analysis down to the No. 100 size.

Three 6 by 12-in. cylinders were molded from each sample. These were tested for compressive strength at 7 days under standard conditions.[10]

SAMPLING COMBINATIONS

Several variations in sequence and

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[4] ASTM Method of Test for Slump of Portland Cement Concrete (C 143 - 58), 1958 Book of ASTM Standards, Part 4, p. 718.

[5] ASTM Method of Test for Weight per Cubic Foot, Yield and Air Content (Gravimetric) of Concrete (C 138 - 44), 1958 Book of ASTM Standards, Part 4, p. 726.

[6] ASTM Method of Test for Air Content of Freshly Mixed Concrete by the Pressure Method (C 231 - 60), 1958 Book of ASTM Standards, Part 4, p. 630.

[7] Calif. Method of Test for Proportions of Coarse Aggregate in Fresh Concrete (529-A), *Materials Manual*, State of Calif., Dept. of Public Works, Division of Highways, Testing and Control Procedures, August, 1960.

[8] Bureau of Reclamation Test for Variability of Constituents in Concrete (A Test of Mixer Performance) (Test 26), *Concrete Manual*, U. S. Dept. of Interior, (Sixth Edition), 1955.

[9] Proposed Tentative Method of Test for Cement Content of Freshly Mixed Concrete (Published as information only), ASTM BULLETIN, No. 239, July 1959, p. 48.

[10] ASTM Method of Test for Compressive Strength of Molded Concrete Cylinders (C 39 - 59), 1958 Book of ASTM Standards, Part 4, p. 655.

number of samples per batch were employed to indicate which combinations might be best adapted to evaluating adequacy of mixing:

1. In batches 1 through 11 and 16 through 25, samples were secured at three points, after approximately $\frac{1}{6}$, $\frac{1}{2}$, and $\frac{5}{6}$ of discharge. The entire routine of tests was performed, as already described, with all except slump being made in the laboratory.

2. Testing procedure was modified in batches 12 through 15 to investigate test reproducibility for closely spaced samples. Three samples were secured as nearly at one time as possible from the same portion of discharge. Testing procedure was identical to that in (1) above.

3. In batches 26 through 33, samples were secured at only two points, after about $\frac{1}{6}$ and $\frac{5}{6}$ of discharge, but centrifuge and drying tests were made in triplicate

TABLE I.—SUMMARY OF DATA FOR BATCHES SAMPLED AT THREE LOCATIONS. Laboratory Test Procedures Emphasized.

Batch	Slump, in.		Air, per cent		+ No. 4 Aggregate per cent Weight of concrete		Air-Free Weight of Mortar, lb per cu ft		Cement by Centrifuge, bags per cu yd		Water by Oven Drying, per cent weight mortar		7-day Compressive Strength		
													psi		Range, per cent
	Avg.	Range	Avg.	Range	Avg.	Range	Avg.	Range	Avg.	Range	Avg.	Range	Avg.	Range	
BATCHES WITH NO WATER ADDED DURING DISCHARGE															
No. 1......	7.1	0.4	1.6	0.1	44.5	5.5	140.4	0.3	5.3	0.5	14.5	0.2	3232	134	4.2
No. 2......	7.8	1.0	4.9	0.0	47.7	4.3	140.3	1.0	5.0	0.1	13.8	0.4	3023	181	6.0
No. 3......	8.1	0.2	5.5	0.5	40.6	6.8	139.5	1.1	5.7	0.4	14.5	0.7	2794	237	8.5
No. 4......	7.3	0.4	3.9	0.2	41.4	2.2	140.5	2.5	6.0	0.9	13.8	0.3	3509	598	17.0
No. 5......	8.6	0.7	3.8	0.1	37.6	2.4	135.8	1.6	6.0	0.1	16.3	0.7	2085	190	9.1
No. 8......	5.9	1.9	6.8	0.8	44.5	2.2	139.5	0.4	5.0	0.6	13.8	0.5	2207	429	19.4
No. 9......	4.3	1.1	1.6	0.1	44.4	1.2	138.5	0.2	6.1	0.2	16.2	0.5	4014	25	0.6
No. 10......	5.7	0.8	1.5	0.2	41.0	9.8	138.3	1.4	7.6	0.3	15.8	1.4	4499	232	5.2
No. 11......	2.9	1.1	2.5	0.2	44.1	2.3	139.7	1.1	5.4	0.1	13.8	0.2	3289	107	3.2
No. 12[a].....	2.6	0.7	1.4	0.2	50.9	2.0	137.3	1.6	5.6	0.2	...	...	4001	76	1.9
No. 13[a].....	2.9	0.8	3.4	0.3	39.1	3.2	142.6	0.4	6.6	0.6	12.5	0.2	3860	75	1.9
No. 14[a].....	2.8	0.8	1.8	0.5	45.1	0.6	137.1	0.0	5.2	0.1	15.6	0.7	3339	106	3.2
No. 15[a].....	6.5	1.0	1.2	0.3	46.6	0.8	132.6	2.5	5.1	0.1	18.3	0.1	3113	37	0.1
No. 16......	6.0	0.3	0.8	0.2	48.1	0.1	132.8	1.5	4.6	0.2	17.4	0.6	2467	88	3.6
No. 18......	5.2	1.1	1.0	0.0	46.9	2.5	133.0	0.6	4.2	0.3	17.4	0.6	2122	112	5.3
No. 19......	7.9	1.0	0.8	0.1	46.4	3.8	131.5	1.2	4.9	0.2	18.1	0.6	2399	50	2.1
No. 25......	4.3	0.8	1.0	0.2	44.0	2.8	135.4	1.3	5.6	0.4	12.4	0.4	3103	229	7.4
No. 34......	3.1	0.6	1.6	0.3	41.9	2.7	137.9	0.9	5.7	0.4	14.9	1.3	2703	563	20.8
No. 35......	6.5	1.8	0.4	0.1	47.2	3.0	133.6	1.3	5.1	0.3	18.1	0.5	1727	19	1.1
No. 36......	4.2	1.4	0.9	0.3	44.1	4.9	134.3	3.1	5.9	0.7	16.2	1.4	2491	622	25.0
No. 37......	7.2	0.6	0.4	0.2	43.7	2.3	131.7	0.7	5.4	0.3	17.4	0.7	1780	69	3.9
BATCHES TO WHICH WATER WAS ADDED DURING DISCHARGE															
No. 6......	2.0	1.8	5.4	1.4	39.5	1.9	140.8	6.9	5.8	0.6	12.0	1.3	2963	789	26.6
No. 7......	7.5	0.7	4.8	0.2	45.7	2.6	138.9	0.5	5.6	0.4	14.5	0.2	3054	150	4.9
No. 17......	1.6	0.1	1.8	0.3	44.8	2.2	135.5	1.6	4.9	0.2	15.6	1.0	3259	330	10.1
No. 20......	3.1	2.6	1.6	0.6	43.9	2.7	136.5	2.3	4.5	0.2	16.1	1.7	2540	570	22.4
No. 21......	5.1	4.6	1.3	0.2	48.1	0.5	136.5	2.6	6.3	0.2	17.5	0.8	3666	115	3.1
No. 22......	3.8	2.0	1.3	0.2	46.3	6.3	135.8	0.5	6.1	0.9	15.5	0.8	3966	56	1.0
No. 23......	5.5	3.1	0.8	0.4	45.6	1.0	134.4	2.2	5.2	0.2	16.0	1.2	2551	425	16.7
No. 24......	4.8	5.9	1.1	0.7	42.5	3.4	136.4	4.8	5.7	0.6	16.2	2.5	3153	1085	34.4

[a] Samples taken as nearly at same point as possible but tested same as sets taken at widely distributed points.

on each sample to study reproducibility. Otherwise, testing procedures were the same as in (1) and (2) above.

4. In batches 34 through 37, samples were secured from three portions as in (1); however, unit weight and air content were measured in the field, and sampling for the centrifuge and drying tests was done in the field. Strength specimens were molded in cardboard molds and stored at the job for 24 hr. The object was to determine if field sampling and testing would serve satisfactorily for evaluating truck mixers when laboratory facilities are not conveniently at hand.

TABLE II.—SUMMARY OF DATA FOR BATCHES SAMPLED AT TWO LOCATIONS. Laboratory Test Procedures Emphasized.

Batch	Slump, in.		Air, per cent		+ No. 4 Aggregate, per cent weight of concrete		Air-Free Wt. of mortar, lb per cu ft		Cement by Centrifuge, bags per cu yd		Water by Oven Drying, per cent weight mortar		7-day Compressive Strength		
													psi		
	Avg.	Range	Avg.	Range	Avg.	Range	Avg.	Range	Avg.	Range	Avg.	Range	Avg.	Range	Range, per cent
BATCHES WITH NO WATER ADDED DURING DISCHARGE															
No. 26......	3.1	1.1	1.1	0.1	43.3	2.2	137.7	0.5	5.9	0.1	15.2	0.2	3301	318	9.6
No. 29......	8.3	0.6	0.4	0.0	40.0	6.4	134.5	0.5	5.9	0.6	16.2	0.5	2466	48	1.9
No. 31......	4.5	1.8	1.4	0.1	42.2[a]	...	136.5[a]	...	5.3[b]	...	15.9	0.1	1977	101	5.1
No. 32......	2.7	0.2	1.5	0.2	44.7	0.4	131.2	0.1	5.2	0.0	14.6	0.0	3035	15	0.5
No. 33......	3.5	0.9	1.0	0.3	48.3	1.0	128.5	1.2	4.3	0.3	15.7	0.1	1715	184	10.7
No. 42......	5.4	0.7	3.9	0.2	46.8	1.1	136.9	0.2	6.0	0.1	16.8	0.1	3369	58	1.7
No. 45......	6.4	0.3	1.5	0.0	46.8	2.2	134.5	0.6	5.1	0.1	16.9	0.3	2136	103	4.8
No. 46......	2.7	0.2	2.1	0.2	41.9	1.3	139.0	0.1	4.7	0.0	14.1	0.0	3204	13	0.4
BATCHES TO WHICH WATER WAS ADDED DURING DISCHARGE															
No. 27......	5.8	2.6	0.6	0.3	48.4	2.4	132.7	1.4	4.5	0.2	17.1	1.3	1687	275	16.3
No. 28......	4.8	2.3	0.9	0.4	45.1	2.2	136.8	1.7	5.6	0.1	15.8	1.2	3021	597	19.8
No. 30......	8.2	0.9	0.4	0.2	42.7	0.3	133.6	3.9	5.6	0.2	17.0	0.6	2082	525	25.2
No. 43......	5.4	2.1	0.8	0.0	48.3	4.2	133.6	1.3	5.0	0.2	18.6	1.9	2842	111	3.9
No. 44......	4.1	0.7	1.2	0.1	50.2	0.6	136.9	0.9	5.5	0.7	16.0	0.2	3302	661	20.0

[a] Single value; no determination on second sample.

[b] Single value; coarse aggregate correction data not secured.

5. In batches 38 through 41, twelve samples were secured, evenly distributed throughout the discharge. Because of the large number of tests, ball penetration was substituted for slump as a measure of consistency, and no strength tests were made. The object was to secure a continuous profile of changes in characteristics throughout the batch.

6. From batches 42 through 46, two samples were secured at about the $\frac{1}{6}$ and $\frac{5}{6}$ points. The usual tests were made, with accent on the laboratory approach, but the centrifuge and drying tests were performed in duplicate to check reproducibility.

7. For batches 47 through 51, the sampling and testing were the same as in (6) above, but field procedures were emphasized.

DISCUSSION OF DATA

To conserve space and avoid duplication, only the data considered to be most indicative of concrete uniformity are presented in tabular form. Average results and within-batch ranges of the following tests are presented in Tables I to IV: slump, air content, per cent of coarse aggregate, air-free unit weight of mortar, cement content by the centrifuge method, water content by drying, and compressive strength. The data are separated to indicate the number of samples tested from the batch, whether or not water was added for retempering between samples, and whether laboratory or field procedures were emphasized in the testing.

TABLE III.—SUMMARY OF DATA FOR BATCHES SAMPLED AT TWO LOCATIONS.
Field Test Procedures Emphasized.

Batch	Slump, in.		Air, per cent		+ No. 4 Aggregate per cent weight of concrete		Air-Free Wt of Mortar, lb per cu ft		Cement by Centrifuge, bags per cu yd		Water by Oven Drying, per cent weight of mortar		7-day Compressive Strength		
													psi		Range, per cent
	Avg.	Range	Avg.	Range	Avg.	Range	Avg.	Range	Avg.	Range	Avg.	Range	Avg.	Range	
BATCHES WITH NO WATER ADDED DURING DISCHARGE															
No. 47......	3.8	0.1	1.1	0.2	48.1	4.0	134.7	1.0	6.3	0.6	16.6	0.0	3854	2	0.5
No. 48......	5.4	0.7	0.6	0.1	51.6	4.7	134.0	2.1	6.1	0.7	16.0	0.2	4056	68	1.7
No. 49......	7.4	0.4	1.3	0.2	47.9	1.0	136.8	0.2	6.3	0.2	16.0	0.4	3650	256	7.0
No. 51......	8.0	0.4	0.8	0.0	49.1	5.6	137.3	2.2	5.2	0.7	16.8	0.0	2861	162	5.7
BATCHES TO WHICH WATER WAS ADDED DURING DISCHARGE															
No. 50......	8.1	1.8	1.2	1.1	45.4	0.9	136.4	3.8	6.2	0.2	16.4	2.0	3481	862	24.8

Table I shows results for the batches from which three samples were taken. Table II gives similar data for batches represented by two samples, when all testing possible was done in the laboratory. Table III also represents 2-sample tests, but with emphasis on performing as many testing operations in the field as possible. Table IV gives the data for individual samples from the four batches which were tested at 12 uniformly distributed points throughout the discharge.

The tabulated data, as well as several additional measured characteristics of the concrete, are shown in graphical form in Figs. 1 to 8. Figure 1 shows ranges in the measured characteristics of concrete from those batches which were represented by tests of three samples—from the early, middle and late portions of discharge. Similar information is given in Fig. 2 for batches represented by two samples—taken near the beginning and end of discharge. Each diagram is divided into two portions, distinguishing the batches to which water was added from those which were not retempered during discharge.

Strength Tests:

Figures 1 and 2 indicate that within-batch variations in strength were small for most batches to which no water was added between samples, but were larger for those retempered. Strength range exceeded 10 per cent for only 5 of 33 undiluted batches, while 10 of 14 batches to which water was added showed strength differences greater than 10 per cent.

Some idea of the significance of the strength differences can be developed by analyzing the uniformity within the sets of three cylinders representing each sample. In this project, since the different samples from a batch were processed at almost the same time and were treated the same as nearly as feasible, it should be possible to distinguish real differences between samples from those due to chance. The program included 123 sets of three cylinders. The average range in strength between highest and lowest of a set was 2.7 per cent of the average. This corresponds to a coefficient of variation of 1.6 per cent for single cylinders from the same population ($v = 0.5907\bar{R}$, where $\bar{R}$ is the average range of sets of three).[11]

This value for coefficient of variation can be used to estimate how large the strength difference between samples must

TABLE IV.—SELECTED DATA FOR BATCHES SAMPLED AT TWELVE LOCATIONS.

Portion of Batch	Ball Penetration, in.	+ No. 4 Aggregate, per cent weight concrete	Cement by Centrifuge, bags per cu yd	Water by Oven Drying, per cent weight mortar	Ball Penetration, in.	+ No. 4 Aggregate, per cent weight concrete	Cement by Centrifuge, bags per cu yd	Water by Oven Drying, per cent weight mortar
	BATCH 38: 4.5 CU YD IN 5 CU YD MIXER; CEMENT 7.0 BAGS PER CU YD				BATCH 39: 5 CU YD IN 5 CU YD MIXER; CEMENT 5.0 BAGS PER CU YD			
1	1.8	43.50	7.21	15.77	3.2	37.28	5.28	16.67
2	2.0	37.72	7.76	15.75	5.0	40.37	5.00	16.86
3	2.2	42.78	6.88	16.23	4.2	42.34	4.75	17.32
4	2.3	46.96	6.47	17.44	3.9	43.16	4.69	16.50
5	2.4	45.64	6.57	16.90	4.3	41.50	4.94	16.90
6	1.0	46.73	6.57	16.07	4.0	43.79	4.54	17.36
7	1.2	46.97	6.46	15.95	4.2	42.42	4.67	18.06
8	2.0	43.78	6.94	16.05	4.3	44.34	4.47	17.73
9	1.8	47.56	6.26	16.77	4.3	45.02	4.33	17.77
10	1.9	48.12	6.36	16.83	4.9	44.74	4.42	18.09
11	2.4	48.05	6.21	16.28	4.7	44.76	4.40	...
12	2.1	42.21	7.00	16.40	4.3	44.18	4.49	17.92
Avg.	1.9	45.00	6.73	16.37	4.3	42.83	4.67	17.38
Range	1.4	10.40	1.55	1.69	1.8	7.74	0.95	1.59
Std. dev.	0.44	3.10	0.45	0.52	0.48	2.26	0.29	0.58
	BATCH 40: 5 CU YD IN 6 CU YD MIXER; CEMENT 6.0 BAGS PER CU YD				BATCH 41: 5 CU YD IN 5 CU YD MIXER; CEMENT 6.0 BAGS PER CU YD			
1	5.2	37.39	6.15	15.84	1.0	41.94	5.38	16.68
2	4.3	33.69	6.50	16.35	1.4	41.85	5.41	15.74
3	4.4	36.88	6.04	16.26	1.9	40.94	5.51	16.21
4	3.9	37.94	6.04	15.77	2.8	42.70	5.34	17.33
5	2.7	38.81	6.05	15.36	2.4	42.81	5.34	16.86
6	3.4	42.97	5.52	16.11	2.8	41.60	5.37	16.47
7	3.0	40.19	5.86	16.33	2.3	43.12	5.32	16.18
8	3.4	40.54	5.77	16.17	2.8	41.66	5.51	16.81
9	3.1	39.38	5.82	15.12	2.8	43.96	5.17	17.04
10	2.9	41.54	5.54	15.55	2.6	37.17	5.92	16.71
11	3.0	41.98	5.60	15.28	2.4	40.80	5.69	17.07
12	2.9	40.85	5.64	15.63	2.5	40.19	5.76	16.41
Avg.	3.5	39.35	5.88	15.81	2.3	41.56	5.48	16.62
Range	2.5	9.28	0.98	1.23	1.8	6.79	0.75	1.42
Std. dev.	0.77	2.58	0.29	0.43	0.59	1.74	0.21	0.44

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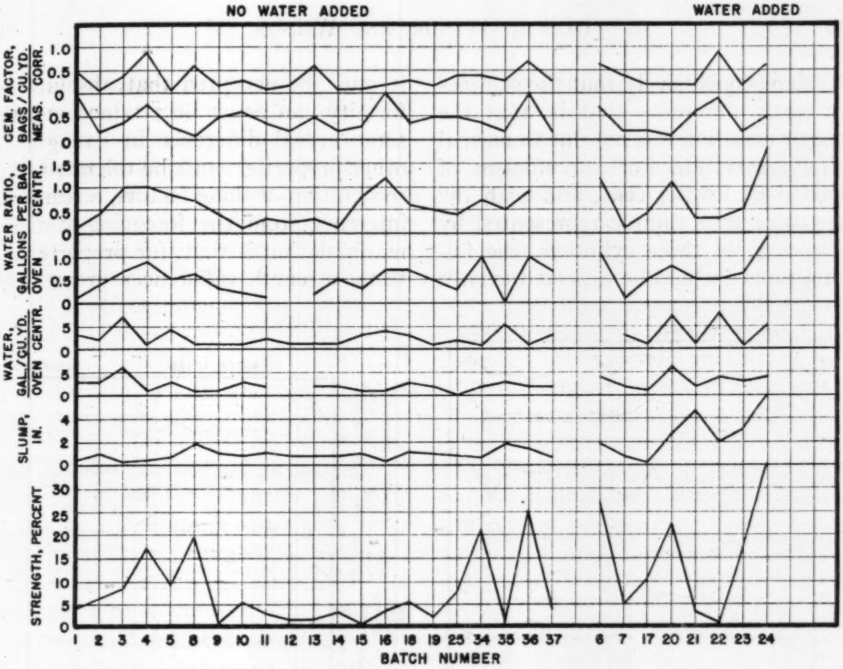
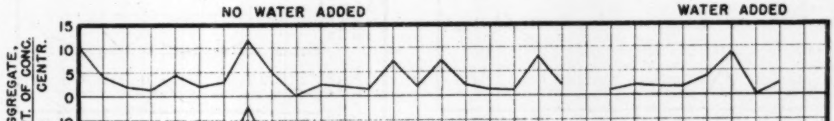
[11] ACI Committee 214 Standard Recommended Practice for Evaluation of Compression Test Results of Field Concrete (ACI 214 – 57), *Proceedings*, Am. Concrete Inst., Vol. 54, p. 1 (1958).



(*a*) Conventional mixture characteristics.



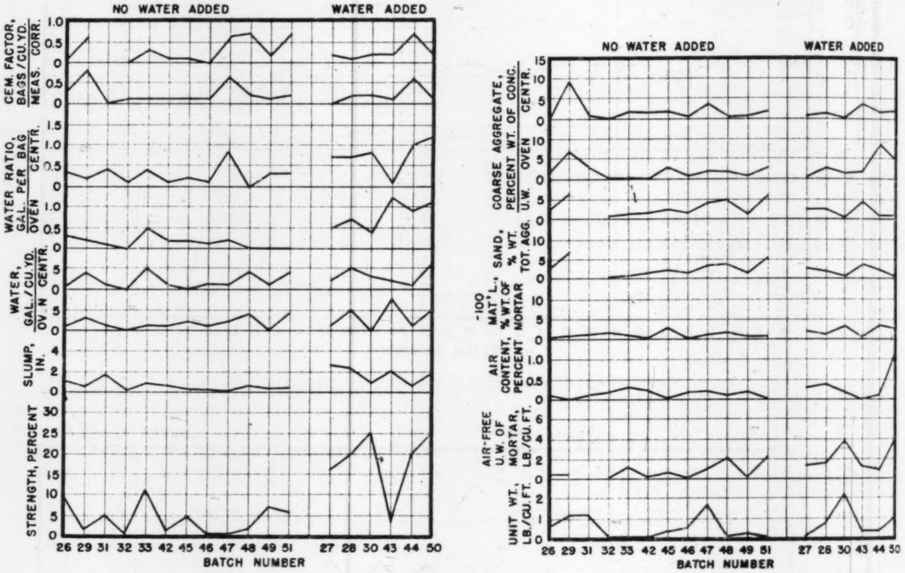
(*b*) Uniformity measures.

FIG. 1.—Ranges of Conventional Mixture Characteristics and Uniformity Measures for Batches Sampled in 3 Portions.

be to show conclusively that the samples were really different—that is, that the strength variation was not due to normal testing errors (**13**). For a coefficient of variation of 1.6 per cent, and with the strength of each sample represented by the average of three cylinders, the following ranges would be expected even if strengths of the different samples were actually the same:

1. For two samples, the difference in strength would exceed 3.4 per cent one in a hundred times.

2. For three samples, the range in strength would exceed 3.9 per cent one in a hundred times.



(*a*) Conventional mixture characteristics.

(*b*) Uniformity measures.

FIG. 2.—Ranges of Conventional Mixture Characteristics and Uniformity Measures for Batches Sampled in 2 Portions.

It should not be concluded that strength ranges in excess of the small amounts discussed above are proof in themselves of unacceptable uniformity. Inevitably, sampling errors will exist no matter how uniform the batch. Further, it must be accepted that absolute uniformity can never be attained and that some actual difference in strength and other properties must be tolerated. With recognition of these factors, a reasonable inference from the foregoing statistical manipulation is that, for properly made tests, strength differences greater than 5 per cent between different portions of a batch can probably be assumed to be actual. Batches exhibiting a strength range of 10 per cent or more would almost certainly have to be considered unacceptably variable.

Slump Tests:

Striking evidence of the ability to measure the effects of added water on the concrete is given by the slump test data in Figs. 1 and 2. When no water was added, slump differences between different portions of a batch never exceeded 2 in. For batches to which water was added between samples, the range of 2 in. in slump was exceeded at least half the time.

Analyses of test reproducibility, similar to those made above for strength, are available for slump. In most cases, slump tests were made in duplicate from each sample. For 100 such pairs of tests, the average difference between the two was 0.5 in. This corresponds to a standard deviation of 0.44 in. $(0.8865\bar{R})^{11}$ and indicates that a difference greater than 1.6 in. will be exceeded by chance alone only about one in a hundred times. If a slump result is taken as the average of two tests, there is only one chance in a hundred that an accidental difference will exceed 1.1 in. By the same reasoning outlined above for strength, some freeboard must be allowed for sampling error and real, but, unavoidable, variations in concrete, suggesting that a difference greater than 2 in. between portions of the same batch should justifiably be considered excessive.

It is of interest that large variations in slump and in strength were not necessarily common to the same batches. If we were to assume that a difference of either 10 per cent in strength or 2 in. in slump were evidence of unsatisfactory uniformity, then the two tests in combination would have detected the effect of the added water in 13 of 14 cases. For the 33 batches to which no extra water was added, lack of uniformity under this double-barreled criterion occurred only for the five which failed the strength limit.

Drying Tests:

Water content derived from either the centrifuge test (extended to include preservation and drying of the coarse as well as fine fractions) or the oven-drying test appeared to correlate in a general way with known additions of mixing water. However, variability of batches to which water was added was only slightly greater on the average than for the others.

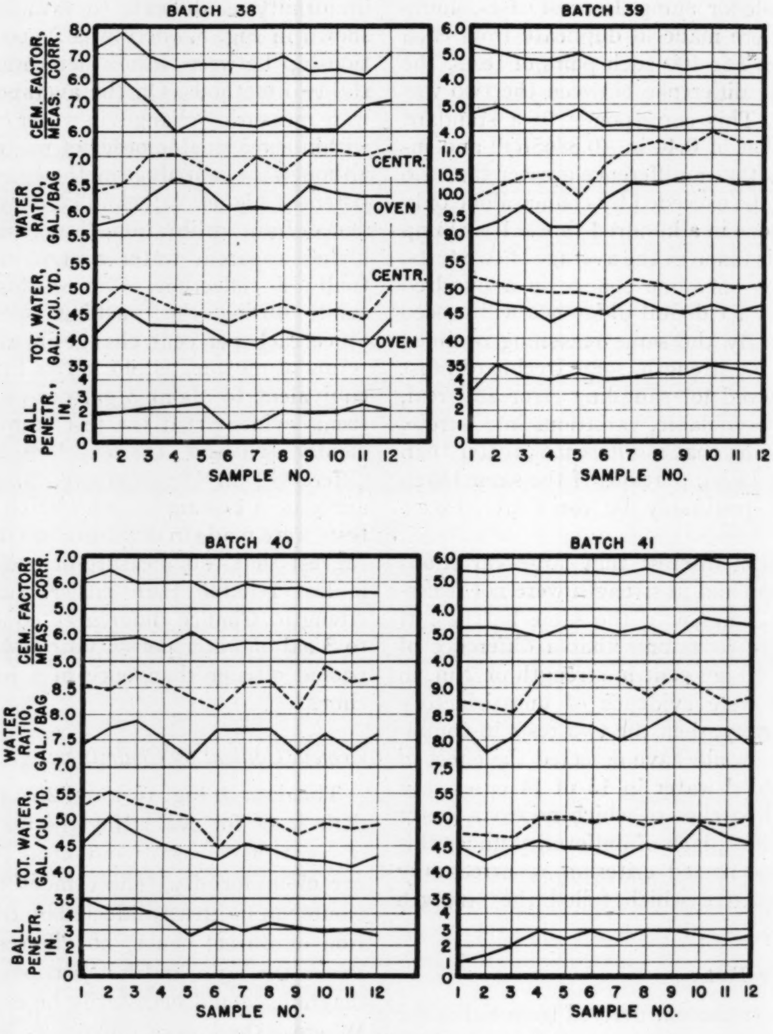
The significance of the water content measurements as criteria of concrete uniformity is difficult to evaluate. As shown in Fig. 5, there was a good relationship between values determined by the two methods. On the average, both were measuring changes in water content within a reasonable order of magnitude, although the centrifuge method gave consistently higher values. There was evidence from calibration tests that this higher apparent water content was actually a reflection of small losses of solids during the washing operation. Since each per cent change in water in terms of mortar, as plotted in Fig. 5, is equivalent to about 3 gal per cu yd, it seems evident that the test is not sufficiently sensitive to reveal significant differences in the concrete. Statistical analyses of 17 samples upon which drying tests were made in duplicate or triplicate suggest that this measure of uniformity is less reliable than either slump or strength. Chance differences equivalent to 3 gal of water per cu yd could be expected oftener than once in a hundred times.

Cement Content by Centrifuge:

The data in Fig. 1(*a*) and 2(*a*) provide little basis for evaluating the usefulness of centrifuge cement content as a measure of uniformity. The cement content would not be greatly affected by the addition of water; hence, no difference between the right and left portions of the diagrams would necessarily be expected. Whereas the known differences in water provided a logical basis for evaluating differences in strength and slump, the utility of the centrifuge test must be assessed on some other basis.

Recently published data (12) have shown that centrifuge tests meticulously made on a given sample, properly corrected for the inevitable sampling errors in coarse aggregate content of the small test portions, are highly reproducible. Standard deviations for single tests of the same sample of laboratory mixed concrete were about 0.1 bag per cu yd.

In the field tests for which data are presented in this report, analyses for the 17 samples upon which replicate determinations were made also showed a within-sample standard deviation of about 0.1 bag per cu yd. This means that an accidental difference as large as ½ bag per

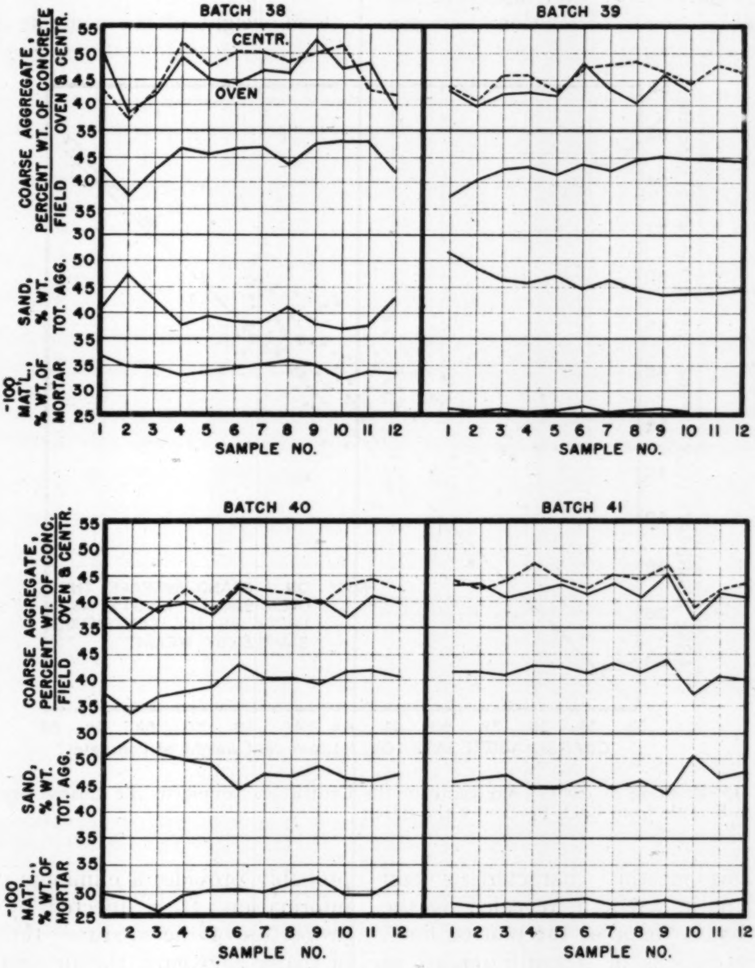


(a) Conventional mixture characteristics.

FIG. 3.—Conventional Mixture Characteristics and Uniformity Measures

cu yd between two tests of the same concrete would be expected to occur only about once in a hundred times.

This evidence of satisfactory reproducibility (but not accuracy) suggests that the centrifuge test will provide a good indication of cement distribution. Actually, with necessary corrections for coarse aggregate sampling error, the test in effect measures cement distribution only in the mortar phase. In view of the large amount of meticulous work and refined calculation involved, it can hardly be considered a preferred method for detecting within-batch variations. As indicated later, equally revealing information can be secured by easier methods.



(b) Uniformity measures.

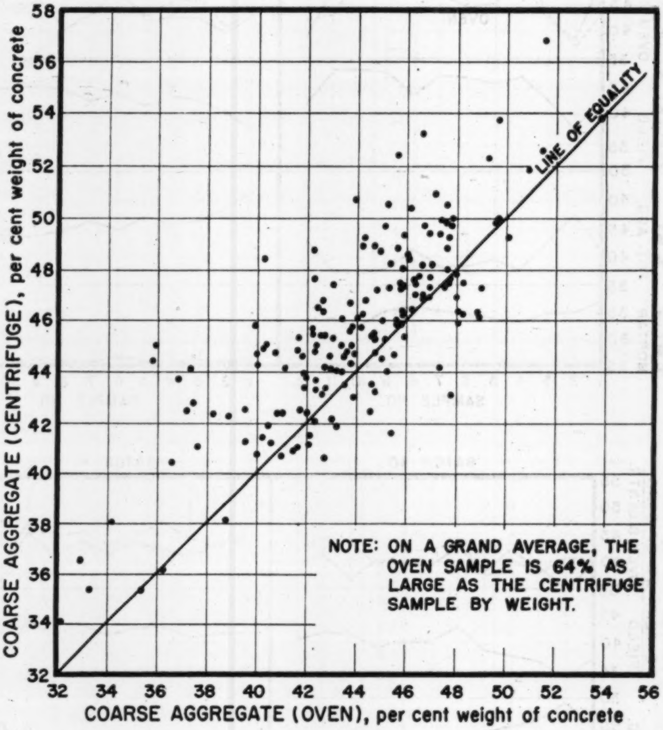
for Multiple Consecutive Samples from Large Batches of Concrete.

Water-Cement Ratio:

Combining the data on mixing water content from the drying tests with cement content by centrifuge permits calculation of a value for water-cement ratio. Figures 1(*a*) and 2(*a*) suggest reasonably good correlation between variations in this characteristic and variations in strength, revealed by the similarity in shape of the plotted lines. High variations in strength tended to accompany high variations in measured-water-cement ratio. More will be said later about general water-cement ratio-strength relationships. In any event, the data indicate that the measurements of either strength or water-cement ratio were sufficiently sensitive to detect real differences in the concrete. For equal yield of information, it appears that the strength tests are preferable from the standpoint of simplified procedure.

Unit Weight Tests:

Unit weight of the concrete in itself probably provides a minimum of useful information. It is affected by several properties of the mixture—the amount of coarse aggregate, the air content, the amount of water, and the proportions of sand to cement. Therefore, variations are difficult to interpret as to cause or significance. Although the data in Figs. 1(*b*) and 2(*b*) show some evidence of greater unit weight variation for batches to



NOTE: ON A GRAND AVERAGE, THE OVEN SAMPLE IS 64% AS LARGE AS THE CENTRIFUGE SAMPLE BY WEIGHT.

FIG. 4.—Relation of Coarse Aggregate in the Centrifuge Sample to the Coarse Aggregate in the Oven Sample from Each Portion of the Batch.

which water was added, it appears that other tests will provide more definitive information on variations in specific characteristics of the concrete.

Some of the complications of the unit weight test can be eliminated by reducing it to terms of air-free mortar.[8] The weight and solid volume of coarse aggregate and the volume of air are eliminated, leaving the density in terms of mortar constituents exclusive of air. Excessive variations in that property reflect changes in water or in proportions of cement and sand. There is evidence in the diagrams that the test did reflect variations caused by the addition of mixing water. If water alone is varied, and the proportions of sand to cement remain the same, a difference in air-free unit weight of mortar of 1 lb per cu ft corresponds to a change in water of about 2 gal per cu yd in the opposite direction.

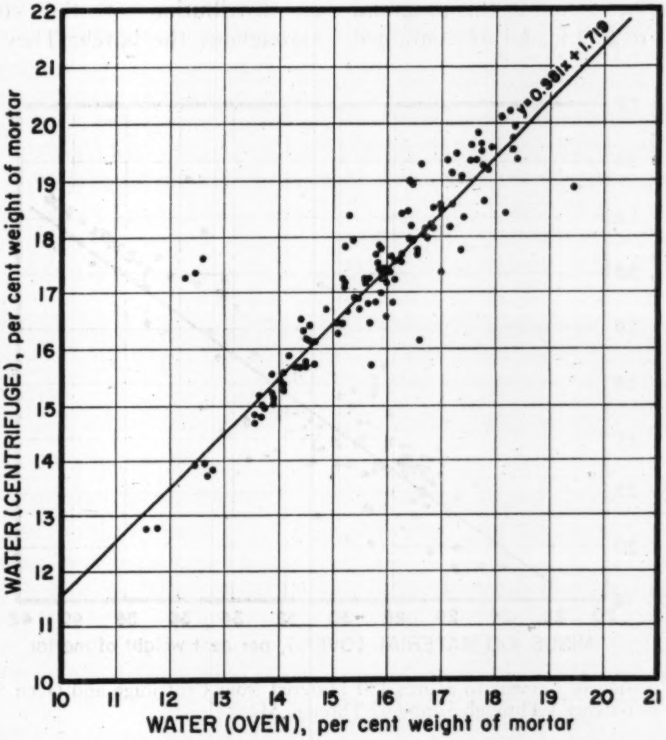


FIG. 5.—Relation of the Percentage of Water from Centrifuge and Oven Samples Taken from Each Portion of Batches 1 Through 37 and 42 Through 51.

No replicate unit weight tests on the same sample were made in this investigation and, therefore, statistical examination of the reproducibility of the test for air-free unit weight of mortar is not possible. However, experience in the laboratory has shown that concrete unit weight measurement is easily reproducible to a degree such that a difference in air-free unit weight of mortar in excess of about 1 lb per cu ft is almost certain indication of a real difference in proportions of the mortar ingredients. With proper regard for problems of sampling and the fact that some real differences in composition are sure to exist in all concrete, a range in air-free unit weight of mortar in excess of about 2 lb per cu ft can probably be considered evidence of unsatisfactory uniformity.

Air Content Measurement:

Of the batches tested in this program, only Nos. 2 to 8, 13, and 42 contained over 3 per cent air and, hence, can reasonably be classed as air-entrained. Of these, only batch No. 6 showed a range in air content larger than 1 per cent. One non-air-entrained batch, No. 50, showed a range of 1.1 per cent with measured values from two locations of 0.6 and 1.7 per cent. On this limited information, no evaluation of air content as a measure of uniformity is justified. However, the test is essential to calculation of the air-free unit weight of mortar. Further, in view of the importance of entrained air to concrete quality, and the ease with which it can be measured, it would seem that this test should be included in any program of uniformity evaluation.

Coarse Aggregate Content:

Ranges in coarse aggregate content from wash-out tests show the uniformity of distribution of the coarse sizes throughout the batch. They should be expected also to provide evidence of variations in harshness of the concrete. The upper three lines of Figs. 1(*b*) and 2(*b*) show within-batch differences as determined by three methods of measurement—separation from the small centrifuge and drying test samples and from the larger, more representative, unit weight sample. It is believed that the latter, because of its larger size, best represented the portion of the batch under consideration. The former two provided information on the reliability of

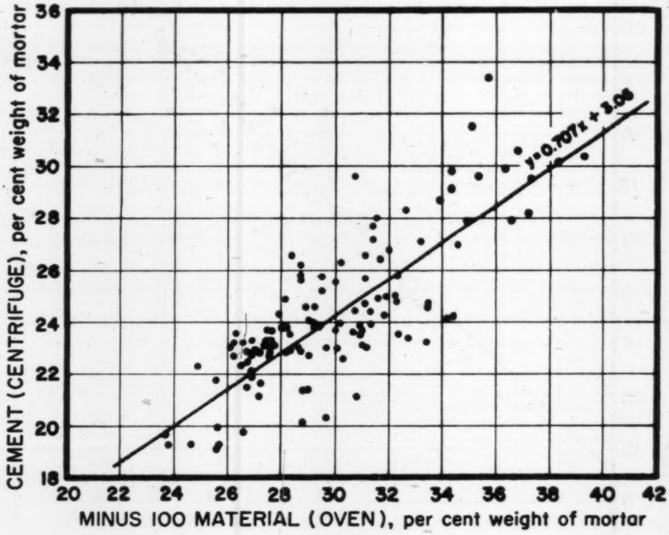


FIG. 6.—Relation of Cement to Minus 100 Material from Centrifuge and Oven Samples from Each Portion of Batches 1 Through 37 and 42 Through 51.

the small size samples used in the centrifuge and drying tests and furnished data for making corrections in results from those tests.

An idea of the significance of variations in amount of coarse aggregate can be obtained from the fact that the difference in workability required for typical structural concrete and that for massive construction or pavement corresponds to a difference in coarse aggregate content of about 3 per cent by weight of the concrete. With allowance for the possibility of errors in sampling and testing, it would seem that a measured difference of more than 5 in the percentage of coarse aggregate by weight of concrete should be considered evidence of lack of satisfactory uniformity. Of the 47 batches for which data are shown in Figs. 1 and 2, six showed coarse aggregate variations higher than this somewhat arbitrary criterion.

Miscellaneous Tests:

Sieve analyses of the drying test samples, which permitted calculation of the per cent of sand and of minus No. 100 material in the concrete, appear to yield a disproportionately small amount of information in relation to the testing complications introduced. As shown in Fig. 6, the minus No. 100 material is related in a general way to centrifuge cement content. However, since other factors such as grading and amount of sand, or of fines from aggregate degradation during mixing, would influence the amount of minus No. 100 sizes, the significance of that determination is difficult to evaluate.

Figures 1 and 2 clearly show that percentages of sand determined from the drying test samples reflect almost precisely the more easily measured variations in coarse aggregate content. This is to be expected since calculation of the sand percentage requires use of the value for coarse aggregate content. It appears, therefore, that the determination of sand percentage provides little useful information which is not more readily available elsewhere.

BATCH "PROFILES"

From each of four batches of concrete, Nos. 38 to 41, twelve samples were secured, evenly distributed throughout the discharge. The scope of tests had to be limited because of the large number of samples, but interesting data on ability to measure trends within the batches were secured. The results are summarized in Table IV and shown graphically in Fig. 3. The diagrams show the actual values for measured characteristics rather than ranges as in the earlier figures.

Figure 3(*a*) suggests a trend in plasticity for batch No. 40, with ball penetration decreasing from beginning to end of discharge. Water content measurements followed the same pattern. For the other three batches, variations in consistency and water appeared random with no very good correspondence between the two.

Both batches Nos. 38 and 39 showed a tendency for water-cement ratio to be lower near the beginning than near the end of discharge. This can probably be attributed to a slight concentration of cement near the discharge end of the mixer, evidenced by higher measured cement factors in the earlier samples. It may be that, with cement entering the mixer in the later stages of the batching cycle, complete and absolutely uniform intermingling with the aggregate was not being achieved.

While the water-cement ratios were always higher for the centrifuge than for the drying test samples (Fig. 3(*a*)) in view of the consistently higher water content measurements in the former method, both methods showed comparable changes and trends, suggesting that random deviations reflected problems of securing representative samples from the mixer rather than of the testing itself.

The distributions of solid ingredients, shown in Fig. 3(*b*), appear consistent with the evidence of Fig. 3(*a*). In batches Nos. 38 and 39 the early portions of discharge contained less coarse aggregate than the later portions, based on the fairly large "field" samples (about 60 lb of concrete). This agrees with the trend noted above toward slightly higher cement in the earlier part of discharge. For all batches, naturally, the sand percentage moves consistently in the opposite direction from coarse aggregate content.

The amounts of coarse aggregate measured in the small centrifuge and drying test samples correspond in a general way with those from the 60-lb "field" sample. They tend to be more erratic, confirming the earlier suggestion that such small samples cannot be representative. The measure of minus No. 100 material appeared quite uniform, but its significance is obscure. It would be expected to correlate, at least roughly, with cement factor, but did not seem to.

The procedure employed here of testing a large number of closely spaced samples is probably too involved for routine checking of within-batch uniformity. However, for research purposes or for establishing over-all mixing requirements and capacities, the method offers promise.

GENERAL RELATIONSHIPS

Interrelations among the various tests provide one means of evaluating their significance and utility. Figures 4 to 8 present such data.

As shown in Fig. 4, coarse aggregate contents determined on the small centrifuge and drying test samples, which should have been the same if the samples had been truly representative, differed considerably. The drying test (oven) sample tended to contain less coarse aggregate than the slightly larger centrifuge test sample. There was evidence that the higher drying temperature in the former method (550 F as opposed to 225 F) may have caused some breakup of coarse particles, causing a slight decrease in material retained on the No. 4 sieve. Differences of the magnitude shown by the spread of points in Fig. 4 would cause errors in measured cement content of 20 per cent or more even if the method of measuring the cement itself were exact.

It should be observed that the nonrepresentativeness of coarse aggregate in small test samples, illustrated in Fig. 4, need not detract from the utility of the centrifuge test for measuring *uniformity* of cement content. A correction can easily be made by measuring the coarse aggregate in the centrifuge sample and adjusting the cement factor accordingly, either on the basis of coarse aggregate put into the batch or its measurements on a larger sample, as was done here. The corrected cement content is calculated as follows:

$$F_c = \frac{F_m(100 - C_a)}{100 - C_s}$$

where:

F_c = corrected cement factor in bags per cu yd or other appropriate units,

F_m = cement factor measured in centrifuge test,

C_a = coarse aggregate content, per cent by weight, actually batched or determined on an acceptably representative sample, and

C_s = coarse aggregate content, per cent by weight, of centrifuge test sample.

It is evident that, for purposes of comparison, cement content expressed in terms of mortar is just as revealing as the corrected value in terms of concrete, when information on coarse aggregate distribution is secured separately.

Figure 5, already mentioned in connection with discussion of within-batch comparisons among concrete characteristics, shows that water contents measured on the centrifuge sample and by the drying test were quite closely related. The former gave measurements, in terms of weight of mortar, which were about 1.5 per cent higher than those from the drying test. Although the relationship is good, it may be deceiving with regard to the utility of the tests for detecting within-batch variations. Test differences of the magnitude suggested by the spread in points of Fig. 5 would be equivalent to as much as 8 gal of water per cu yd (even disregarding the few widely divergent points). This is far beyond the error which could be tolerated in making within-batch comparisons.

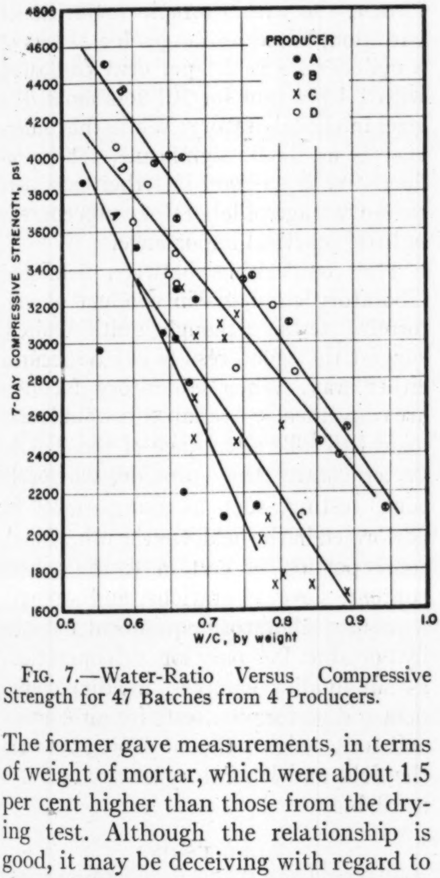


FIG. 7.—Water-Ratio Versus Compressive Strength for 47 Batches from 4 Producers.

As would be expected, there was a general relation between cement as determined in the centrifuge test and the minus No. 100 sieve fraction extracted from the drying sample (Fig. 6). The scatter of points is such as to suggest that one or both of the methods was insufficiently precise to provide reliable checks on uniformity of concrete. As mentioned earlier, the minus No. 100 sieve size includes material other than cement and would not necessarily give a consistent indication of cement content.

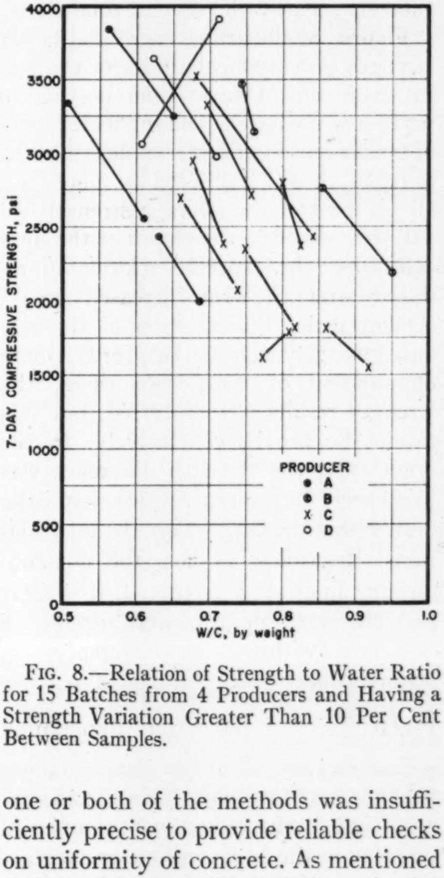


FIG. 8.—Relation of Strength to Water Ratio for 15 Batches from 4 Producers and Having a Strength Variation Greater Than 10 Per Cent Between Samples.

Figures 7 and 8 show relationships between water-cement ratio and strength. Average points for individual batches, in Fig. 7, yield quite well defined curves for each producer. Apparently the measurements of water-cement ratio were accurate enough on the average to relate well to strength. Perhaps of more significance is the evidence that strength will yield as good information on changes in water-cement ratio as the more involved direct measurement. The different levels of the curves in Fig. 7 can be attributed to differences in materials, particularly cement, used by the several producers.

Figure 8 illustrates the ability of strength measurement to detect changes in water-cement ratio from portion to portion of the batch. The connected pairs of points represent test samples from the same batch which differed by more than 10 per cent of the average strength. In only two cases do the slopes of the lines contradict the expected relation of reduced strength with increased water-cement ratio. Examination of the raw data strongly suggests that, for the more outstanding of those two cases, the strength results were reversed, but this cannot be proved conclusively. In the other case, the strength difference was just barely 10 per cent, which may have been within the range of accidental variations. Regardless of how the non-conforming lines are interpreted, it is clear that the strength tests were capable of detecting within-batch differences in water ratio.

FIELD VERSUS LABORATORY PROCEDURE

Examination of within-sample ranges for the centrifuge and drying tests suggests that careful reduction of samples to test size in the field is about as satisfactory as performing all possible sample reductions in the laboratory. The number of cases with replicate tests on a given sample were too few to provide for statistical analyses. However, for ten pairs of drying tests on field-secured samples, the average range in water content was actually slightly lower than for ten similar pairs removed from composited samples in the laboratory. In the case of the centrifuge test, the average within-sample range in cement was higher for field-secured samples, but the difference was due entirely to a single apparently erratic value.

When strength test cylinders were molded in cardboard molds and left on the job the first day, results were less reproducible than for specimens fabricated in the laboratory in steel molds. For the former, the within-sample coefficient of variation, based on ranges for 22 sets of 3 cylinders, was 2.4 per cent compared with 1.4 per cent for 101 sets molded in steel in the laboratory. While this difference is no doubt significant, the reproducibility is so good in either case that the advantage of laboratory procedure is of little practical importance.

The comparisons between field and laboratory emphasis in conduct of uniformity tests, although quite limited, suggest that good results can be secured either way. When laboratory facilities are conveniently available, it would seem logical to take advantage of the slightly better control they provide. Obviously some testing, such as slump, must be performed in the field. On the other hand, some portion of certain tests—cement content, sieve separations, and strength—require laboratory equipment not easily portable. It is only for such operations as subdividing samples, molding cylinders and performing tests for air content and unit weight, that a choice between the field and laboratory environment is available.

CONCLUSIONS

The investigation demonstrated that a wide variety of tests will provide useful information on within-batch variations in concrete. Some are easily made with ordinary field testing equipment while others involve refined laboratory procedures and meticulous care on the part of skilled personnel. The selection of a program of sampling and testing will depend upon the object for which uniformity is being evaluated.

The program described was directed primarily to establishing a basis for evaluating the efficiency of truck mixers. Such a procedure is needed for checking required amount of mixing, establishing capacities, detecting blade wear, and evaluating batching sequences. For such purposes, it appears that a minimum routine should involve two samples, carefully taken to be representative of early and late portions of the discharge — probably about the $\frac{1}{6}$ and $\frac{5}{6}$ points. At the least, each sample should be tested for slump, weight per cubic foot, air content, percentage of coarse aggregate by wash-out test, and compressive strength. These tests are easily made and involve a minimum of special refinement to usual concrete testing techniques. They provide information on the important characteristics which should be maintained uniform: consistency, workability, air content (as evidence of potential durability), water content, and strength.

The following limits appear to define the approximate maximum differences likely to occur in reasonably uniform batches. Greater differences can be interpreted as evidence of unacceptable uniformity:

1. Slump, 2 in. (With each portion represented by duplicate tests, a difference greater than about 1½ in. should be viewed with suspicion.)

2. Coarse aggregate content, 5 per cent by weight of concrete. (This is about the equivalent of saying, for typical concrete, that the amount of coarse aggregate per unit volume shall not vary more than 10 per cent.)

3. Air content, 1 per cent. (This value is largely intuitive, based on the need for reasonable assurance that air content will be adequate for durability protection.)

4. Compressive strength, 10 per cent.

5. Air-free unit weight of mortar, 2 lb per cu ft. (This is approximately equivalent to the ± 0.8 per cent limit set by the Bureau of Reclamation (17). It is consistent with evidence derived in this study from batches to which water was added and were, therefore, known to have varied in composition. The value can be calculated from the unit weight of concrete, adjusted for coarse aggregate and air contents.)

Additional information can be secured from the centrifuge cement content test and the drying test, although the latter probably adds little to the indications of slump and strength tests. Because of the small size of sample used, with its questionable representation of coarse aggregate, the centrifuge test should be relied upon only to indicate variations in composition of the particular batch of concrete, not as a check on the cement factor itself. The test is quite involved and, in most cases of mixer evaluation, its yield of information would hardly be commensurate with the time and labor required.

Increasing the number of samples from each batch will yield additional information. When that is done, proper recognition should be given to the fact that the incidence of large accidental differences in test results will increase with the number of samples. Therefore, application of the limits suggested above should be made with caution.

The testing procedure outlined should be applicable to the following:

1. Determining amount of mixing required to assure uniformity.

2. Determining the amount of concrete which can be satisfactorily mixed in a given mixer.

3. Establishing efficient mixing speeds.

4. Checking the condition of mixer blades.

5. Determining the efficiency of equipment for introducing water into the truck mixer.

6. Evaluating the efficiency of batching sequences and methods with regard to facilitating mixing.

7. Checking discharged concrete for evidence of segregation.

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(7) A. G. Timms, "Performance Tests of Concrete Truck Mixers," *Proceedings*, Am. Soc. Testing Mats., Vol. 57, pp. 1012–1026 (1957). Reprinted Nat. Ready Mixed Concrete Assn. Publication No. 78, March, 1958.

(8) Glenway Maxon, "Evaluating the Performance of Stationary Concrete Mixers," Presented at the Sixty-Third Annual Meeting of the Am. Soc. Testing Mats., Atlantic City, New Jersey, June 26–July 1, 1960.

(9) I. Narrow, "Performance Tests of Field Concrete Mixers," *Proceedings*, Am. Soc. Testing Mats., Vol. 60, p. 1047 (1960).

(10) W. G. Hime and R. A. Willis, "A Method for the Determination of the Cement Content of Plastic Concrete," ASTM BULLETIN No. 209, October, 1955, p. 37.

(11) D. O. Woolf, "Effects of Time of Mixing on Strength of Concrete in 34-E Dual Drum Pavers," *Public Roads*, Vol. 31, No. 1, April, 1960.

(12) S. Walker, D. C. Bloem, R. D. Gaynor, and J. R. Wilson, "A Study of the Centrifuge Test for Determining the Cement Content of Fresh Concrete," (*to be published*).

(13) Carl A. Bennett and Norman L. Franklin, "Statistical Analysis in Chemistry and the Chemical Industry," John Wiley and Sons Inc., New York, N. Y.

DISCUSSION

Mr. Calvin C. Oleson.[1]—In the report by Bloem, Gaynor, and Wilson, it would appear that the specification that has been approved by Committee C-9 on Concrete and Concrete Aggregates as a Tentative Revision of Specification C 94 is either too restrictive or that different values should be applied to transit mixers than are being applied to central mixers. For central mixers, the Task Force has established that samples should represent the end of the first tenth and the beginning of the last tenth of the batch. The tests by Bloem et al represent the end of the first *sixth* and beginning of the last *sixth*, which has the effect of approaching more closely the middle of the batch. This is usually the portion that is uniform if there is any uniformity at all in the batch.

The report by Bloem, Gaynor, and Wilson offers tolerances in slump, air content, and other controls that are considerably broader than those established for large central mixers. Perhaps time and experience will show that the tolerances given in the revision of Section 9(*c*) are too strict and should be relaxed. Perhaps, too, the portions of the batch that are being compared are too close to the extremes, although that will always remain a matter of opinion with individual engineers.

Mr. J. J. White.[2]—I do not question the worth of suitably prepared and tested concrete cylinders. Undoubtedly, the reports of such tests are the best information available of the concrete, prior to its placement in the structure, as conformance to the requirements of the specifications for proportions, mixing, and transporting. I grant that it may be deduced from such reports that the hardened concrete in the structure will also conform to the requirements of the specifications, but such deductions leave entirely too much to speculation as to the successive steps of suitably constructed and braced formwork, placing, vibrating, curing, and finishing. I am not unmindful of the part that inspection should play in such field work. Admittedly, impact hammer and sonic testing methods are not the last word. In any event, it is my hope that in due time this Society will devote a small part of the interest now directed to testing the compressive strength of concrete cylinders to effective tests of the hardened concrete in place in the finished structure.

Messrs. D. L. Bloem, R. D. Gaynor, and J. R. Wilson (*authors' closure*).—Mr. Oleson observes that tolerances on the uniformity of concrete characteristics suggested by the authors are broader than permitted for central mixers in a tentative revision of ASTM Specifications C 94. In the opinion of the authors, the two sets of limits are not directly comparable. Specifications C 94 establishes criteria which may be used as a basis for waiving arbitrary mixing time requirements. The burden of proof is placed on the mixer to demonstrate its capability of producing a high degree of uniformity; hence the limits are, as they probably should be, quite stringent. They define ranges within which good uniformity can be considered proven beyond reasonable doubt. On the other hand, the variations discussed by the authors were presented as defining maximum limits beyond which uniformity was considered to be unacceptable.

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[2] Parsons, Brinckerhoff, Quade, and Douglas, Engineers, New York, N. Y.

Tolerances mentioned in the paper are in good agreement with criteria suggested by other investigators for use in mixer evaluation. The Bureau of Reclamation[3] uses a range of ±0.8 per cent in air free unit weight of mortar and ±5 per cent in weight of coarse aggregate per unit volume of concrete as maximum permissible variations within a batch. Except for differences in units used in their expression, these are virtually identical to the values stated by the authors.

A recent paper by I. Narrow[4] sets tentative limits for permissible ranges at 1 in. for slump, 20 per cent for volume of air per unit volume of concrete, 8 per cent for coarse aggregate content, and 10 per cent for strength. Here again, except for the lower tolerance on slump, the values are in good agreement with the authors'. A report of the U. S. Army Waterways Experiment Station,[5] issued subsequent to the paper under discussion, suggests limits of ±8 per cent on air-free unit weight of mortar and ±5 per cent on coarse aggregate content. The State of California Division of Highways employs a wash-out test to determine the quantity of coarse aggregate in concrete.[6] A difference of 15 per cent from the batched quantity of sizes larger than $\frac{3}{4}$ in. is considered evidence of objectionable lack of uniformity. That difference, expressed in terms of total aggregate, would be comparable in magnitude to the limits mentioned above.

In summary, published literature supports the authors' suggested bases for distinguishing concrete which is demonstrably non-uniform.

Mr. Oleson also raises the question of whether batches should be sampled at one-tenth or one-sixth of the distance from each end. This would appear to be a matter of judgement. The object is to assure that widely separated points are represented without use of the absolute extremes which, in any mixer, are likely to be non-representative. In any event, for most mixers, the amount of concrete between the one-tenth and one-sixth points is probably too small to be estimated.

The authors agree with Mr. White that strength tests of cylinders do not provide a quantitative evaluation of the structure itself. In the reported test program, the strength tests were merely one of several bases upon which mixing efficiency was evaluated. Uniformity of fresh concrete is a necessary precursor to the high quality in the finished structure sought by Mr. White.

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[3] U. S. Bureau of Reclamation, Concrete Manual (Sixth Edition, 1955), U. S. Government Printing Office, Washington 25, D. C.

[4] I. Narrow, "Performance Tests of Field Concrete Mixers," *Proceedings*, Am. Soc. Testing Mats., Vol. 60, p. 1047 (1960).

[5] U. S. Army Engineer Waterways Experiment Station, "Development of Method of Test for Concrete Mixer Performance," Technical Report No. 6-562, March, 1961.

[6] State of California Department of Public Works, Division of Highways, Method of Test for Proportions of Coarse Aggregate in Fresh Concrete, Method No. Calif. 529-A, Materials Manual, August, 1960.

CONCRETE CONTAINING FLY ASH AS A REPLACEMENT FOR PORTLAND BLAST-FURNACE SLAG CEMENT*

By W. E. Grieb[1] and D. O. Woolf[1]

Synopsis

This paper reports the results of test on the use of fly ash as a replacement for part of the cement in concrete prepared with portland cement and portland blast-furnace slag cement and tests of a recommended adjusted mix containing fly ash. These tests show that fly ash can be used satisfactorily as a replacement for portland cement or portland blast-furnace slag cement, but the properties of the concrete are affected by the carbon content of the fly ash. It is indicated that the amount of fly ash used should be correlated with the amount of lime released by the cement. It also indicates that the adjusted mix, containing a low-carbon fly ash, will give strengths at 28 days equal to similar concrete without fly ash.

A report entitled "Use of Fly Ash in Concrete" was presented at the 1956 Annual Meeting of the Society.[2] That report covered the properties of concrete prepared with four fly ashes as replacements for either $\frac{1}{6}$ or $\frac{1}{3}$ of the volume of the cement. The fly ashes differed widely in carbon content. Two type I portland cements were used, one having a high alkali content and the other a low alkali content.

In the discussion of the 1956 report, several questions were raised regarding the use of fly ash. As a result of these questions and others received at a later date, further tests were made.

One of the questions raised was whether fly ash could be used as a replacement for part of the cement in concrete mixes prepared with portland blast-furnace slag cement. This cement is defined as an intimately interground mixture of portland-cement clinker and granulated blast-furnace slag. The slag content in the final product is limited to 25 to 65 per cent by weight.[3] The finely ground slag is, by itself, only slightly cementitious. Concern was expressed over the possibility that a further dilution of the cement constituent by replacing part of this cement with fly ash might cause the concrete to have unfavorable strength characteristics. Although fly ash has a pozzolanic reaction with lime liberated by portland cement during hydration, there was a question of whether sufficient lime would be available for this reaction.

In the first part of this investigation, tests, identified as series I, were made on concretes prepared with portland cements and with portland blast-furnace slag cements in which $\frac{1}{3}$ of the cement by volume was replaced by fly ash.

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* Presented at the Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] Highway Research Engineers, U. S. Dept. of Commerce, Bureau of Public Roads, Washington 25, D. C.

[2] A. G. Timms and W. E. Grieb, "Use of Fly Ash in Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 56, p. 1139 (1956).

[3] Tentative Specifications for Portland Blast-Furnace Slag Cement (C 205 – 58 T), 1960 Supplement to ASTM Book of Standards, Part 4.

TABLE I.—CHEMICAL COMPOSITION AND PHYSICAL PROPERTIES OF CEMENTS AND FLY ASHES.

	Portland Cements		Portland Blast-Furnace Slag Cements		Fly Ashes	
	B-I	E-I	B-IS	E-IS	B	Y
Chemical composition, per cent						
Silicon dioxide	20.8	21.3	26.2	26.1	49.2	38.5
Aluminum oxide	5.1	5.6	7.8	6.7	19.9	23.5
Ferric oxide	3.1	3.5	2.3	2.4	16.2	18.8
Calcium oxide	63.6	62.5	53.6	55.7	5.5	3.2
Magnesium oxide	2.8	1.7	3.6	2.8	1.4	1.0
Sulfur trioxide	1.8	2.3	2.3	2.2	2.7	0.6
Loss on ignition	1.6	1.3	2.0	2.2	1.2[a]	11.6[a]
Sodium oxide	0.09	0.15	0.09	0.15	2.00	0.60
Potassium oxide	0.15	1.02	0.20	0.91	2.35	1.88
Total equivalent alkalies as Na_2O	0.19	0.82	0.22	0.75	3.55	1.84
Insoluble residue	0.16	0.20	0.73	0.26	...	...
Chloroform, soluble	0.003	0.003	0.006	0.008	...	...
Carbon	...	...	...	...	0.6	11.2
Computed compound, per cent						
Tricalcium silicate	57	43	...	...	...	...
Dicalcium silicate	17	28	...	...	...	...
Tricalcium aluminate	8	9	...	...	...	...
Tetracalcium aluminoferrite	9	11	...	...	...	...
Calcium sulfate	3.1	3.9	...	...	...	...
Slag used in manufacturing, per cent	...	...	45	35	...	...
Physical properties						
Apparent specific gravity	3.12	3.14	3.03	3.03	2.52	2.43
Specific surface, sq cm per g	3325	3555	4820	3605	4305	3220
Passing 325 sieve, per cent	87.5	96.2	97.6	94.9	95.2	89.4
Autoclave expansion, per cent	0.06	0.04	0.00	0.00	...	...
Normal consistency, per cent	23.8	26.5	27.2	26.6	...	...
Time of setting (Gillmore)						
Initial, hr	3.7	2.9	3.4	2.8	...	...
Final, hr	5.6	4.8	6.2	6.2	...	...
Compressive strength, 1:2.75 mortar						
At 7 days, psi	2625	3525	2630	2360	...	...
At 28 days, psi	4150	4965	5190	3900	...	...
Mortar air content, per cent	10.4	9.4	7.4	9.2	...	...

[a] Determination made at 600 C.

The 1956 investigation indicated that a direct replacement of either $\frac{1}{6}$ or $\frac{1}{3}$ of the portland cement by fly ash resulted in reduced strength of the concrete at all ages through 28 days. It was also found that replacement of $\frac{1}{3}$ of the cement by fly ash adversely affected the resistance of concrete to attack by calcium chloride used for ice removal.

In the discussion of the 1956 report, a producer of fly ash recommended the use of an adjusted mix containing fly ash to furnish good early strength and resistance to scaling of concrete. In this mix, the cement content was reduced from 6.0 to 5.25 bags per cu yd, the sand-to-total aggregate ratio was reduced 2 per cent, and an addition of 100 lb (approximately $1\frac{1}{3}$ bags) of fly ash was made. To determine the effect of this mix on the durability and strength of concrete, tests identified as series II were made.

MATERIALS

In this investigation, tests for compressive and flexural strength and for durability were made on air-entrained concrete mixes prepared with two fly ashes and four cements. The fly ashes were similar to those used in the previous investigation, but different cements were used.

The two fly ashes used were identified as B and Y and corresponded to fly ashes B and Y used in the previous investigation. Fly ash B had a low carbon content (0.6 per cent) and fly ash Y had a high carbon content (11.2 per cent). ASTM Tentative Specification C 350 - 60 T[4] limits the loss on ignition of fly ash to 12 per cent, this loss on ignition being due mainly to the carbon content. Both fly ashes met this requirement.

The cements used included a type I portland cement and a type IS portland blast-furnace slag cement from each of two cement plants. The same clinker was used in the manufacture of the two types of cement from the same plant. These cements are identified as cements B-I and B-IS, and E-I and E-IS, and correspond to the cements of the same identification reported in a previous investigation of portland blast-furnace slag cement in concrete.[5] The two cements from source B were low alkali cements and those from source E were high alkali.

The chemical composition and physical properties of the fly ashes and cements are given in Table I.

The aggregates were similar to those used in the previous investigation—a siliceous sand and a crushed limestone. The concretes for strength tests were made with $1\frac{1}{2}$-in. maximum size coarse aggregate, and for durability tests the maximum size was 1 in. A commercially available aqueous solution of neutralized Vinsol resin was used to entrain air.

MIX PROPORTIONS

The mix data for all concrete specimens are given in Table II. All tests were made on air-entrained concrete. The control mixes without fly ash were designed for a cement content of 6.0 bags of cement per cu yd of concrete with a slump of 3 in. and an air content of 5 per cent.

One percentage replacement of fly ash ($33\frac{1}{3}$ per cent) was used for series I. In the 6-bag mix, 2 bags of cement were replaced by an equal solid volume of fly ash. Because of the differences in the specific gravities of the materials, 74 lb of fly ash B or 72 lb of fly ash Y was equivalent to the solid volume of a 94-lb bag of cement. The weight of the fine and coarse aggregates per cubic yard of concrete were the same for the mixes with and without fly ash.

This amount of fly ash is more than would ordinarily be used in highway construction. However, if this amount could be used without detrimental effects, then lesser amounts could also be used.

For the series II tests, the mixes without fly ash were the same as those used in series I. Where fly ash was used, the mix was adjusted as recommended by the producers of fly ash. Three-quarters of a bag of cement (70.5 lb) was replaced by $1\frac{1}{3}$-bags of fly ash (100 lb), and to compensate for the

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[4] Tentative Specifications for Fly Ash for Use as an Admixture in Portland Cement Concrete (C 350 – 60 T), 1960 Supplement to ASTM Book of Standards, Part 4.

[5] W. E. Grieb and George Werner, "Final Report of Tests of Concrete Containing Portland Blast-Furnace Slag Cement," *Public Roads*, August 1961.

TABLE II.—CONCRETE MIX DATA.

Cement			Fly Ash		Mixes for Strength Tests[a]					Mixes for Durability Tests[a]				
Identification														
Source	Type	Amount, bags per cu yd	Identification	Amount, lb per cu yd	Water, gal per cu yd	Slump, in.	Plastic Concrete, lb per cu ft	Air Content, per cent	Air Entrained Admix.,[b] per cent	Water, gal per cu yd	Slump, in.	Plastic Concrete, lb per cu ft	Air Content, per cent	Air Entrained Admix.,[b] per cent
SERIES I														
B.......	I	6.0	None	None	31.2	3.1	146.8	4.6	100	34.4	2.4	145.5	4.9	100
		4.0	B	148	31.0	3.3	146.2	4.7	202	31.3	2.0	144.9	5.8	171
		4.0	Y	144	31.5	3.0	145.4	5.2	617	35.8	2.9	144.7	4.2	500
B.......	IS	6.0	None	None	31.9	2.9	147.2	4.8	100	35.3	2.9	145.5	4.2	100
		4.0	B	148	29.8	2.9	146.8	4.2	104	35.2	2.7	143.5	5.9	129
		4.0	Y	144	31.5	2.7	145.1	4.8	239	34.1	2.6	140.2	6.7	321
E.......	I	6.0	None	None	31.5	2.9	147.4	5.4	100	34.5	2.6	145.5	5.1	100
		4.0	B	148	31.3	2.9	147.5	4.7	138	32.7	2.5	145.5	5.2	157
		4.0	Y	144	32.1	3.0	145.9	5.1	447	35.9	2.6	144.7	4.9	429
E.......	IS	6.0	None	None	31.6	2.8	146.4	5.1	100	35.0	2.8	143.5	5.8	100
		4.0	B	148	30.4	3.3	146.6	4.6	169	33.0	3.2	142.7	5.5	143
		4.0	Y	144	31.7	3.0	146.7	4.2	434	36.6	3.2	142.7	4.5	386
SERIES II														
B.......	I	6.0	None	None	31.2	3.1	146.8	4.6	100	34.4	2.4	145.5	4.9	100
		5.25	B	100	31.3	3.2	147.2	4.5	183	32.8	2.2	146.9	5.0	143
		5.25	Y	100	31.8	3.2	146.1	4.9	488	35.5	2.4	145.1	4.2	357
B......	IS	6.0	None	None	...	...	...	...	...	35.3	2.9	145.5	4.2	100
		5.25	B	100	...	...	...	...	...	34.2	2.0	146.3	4.2	107
		5.25	Y	100	...	...	...	...	...	36.2	2.5	145.7	4.2	214
E.......	I	6.0	None	None	31.5	2.9	147.4	5.4	100	34.5	2.6	145.5	5.1	100
		5.25	B	100	30.6	2.8	146.6	5.1	173	34.8	2.5	145.5	4.4	129
		5.25	Y	100	32.1	3.0	146.7	4.9	334	36.4	3.1	144.5	4.5	286
E.......	IS	6.0	None	None	...	...	...	...	...	35.0	2.8	143.5	5.8	100
		5.25	B	100	...	...	...	...	...	35.2	3.1	143.3	5.5	129
		5.25	Y	100	...	...	...	...	...	37.1	3.0	144.1	4.4	314

[a] Proportions for mixes without fly ash were 94 lb cement; 170 lb sand; 350 lb limestone.

[b] Relative amount of air-entraining admixture used, amount in concrete without fly ash considered as 100 per cent.

greater volume of fly ash as compared with the volume of cement replaced, the sand content was reduced by approximately 45 lb per cu yd. The weight of the coarse aggregate per cubic yard of concrete was the same for the mixes with and without fly ash.

Table II shows that for both series I and II, the mixes prepared with fly ash B generally required slightly less water than was required for the corresponding mix without fly ash. Where fly ash Y was used, slightly more water was generally required than for the mix without fly ash. This was true for both the concretes prepared with the portland cements and for those prepared with the portland blast-furnace slag cements.

The amount of air-entraining admixture required to obtain air contents of 4 to 7 per cent was greater for all mixes containing fly ash replacement than for the corresponding mixes without fly ash. Where fly ash B was used, from 4 to 102 per cent more air-entraining admixture was needed and for fly ash Y the amount increased to 139 to 517 per cent. The relative amounts varied according to the type and source of the cement, the size of the coarse aggregate, and the carbon content of the fly ash. For the mixes without fly ash, more air-entraining agent was required for the concretes prepared with the slag cement than for those prepared with the portland cement.

TABLE III.—RESULTS OF STRENGTH TESTS.[a]

Cement			Fly Ash		Flexural Strength			Compressive Strength			Strength Ratio[b]					
Identification		Amount, bags per cu yd	Identification	Amount, lb per cu yd	7 Days, psi	28 Days, psi	1 Yr, psi	7 Days, psi	28 Days, psi	1 Yr, psi	Flexural Strength			Compressive Strength		
Source	Type										7 Days, per cent	28 Days, per cent	1 Yr, per cent	7 Days, per cent	28 Days, per cent	1 Yr, per cent
SERIES I																
B.......	I	6.0	None	...	585	755	800	3580	5340	6900	100	100	100	100	100	100
		4.0	B	148	420	630	980	2240	4050	7280	72	83	122	63	76	106
		4.0	Y	144	325	550	920	1730	3190	6040	56	73	115	48	60	88
B.......	IS	6.0	None	...	525	750	935	3360	6180	8250	100	100	100	100	100	100
		4.0	B	148	410	695	920	2160	4950	7590	78	93	98	64	80	92
		4.0	Y	144	345	615	900	1660	3850	6990	66	82	96	49	62	85
E.......	I	6.0	None	...	685	780	790	4340	5410	6650	100	100	100	100	100	100
		4.0	B	148	555	780	975	3210	4980	7550	81	100	123	74	92	114
		4.0	Y	144	420	640	950	2380	3950	6290	61	82	120	55	73	95
E.......	IS	6.0	None	...	495	720	935	2680	4460	6450	100	100	100	100	100	100
		4.0	B	148	370	740	920	1930	3750	5670	75	103	98	72	84	88
		4.0	Y	144	290	490	895	1390	2980	4950	59	68	96	52	67	77
SERIES II																
B.......	I	6.0	None	...	585	755	800	3580	5340	6900	100	100	100	100	100	100
		5.25	B	100	515	750	985	3350	5280	8550	88	99	123	94	99	124
		5.25	Y	100	490	705	955	2890	4680	7680	84	93	119	81	88	111
E.......	I	6.0	None	...	685	780	790	4340	5410	6650	100	100	100	100	100	100
		5.25	B	100	630	780	895	3890	5430	7740	92	100	113	90	100	116
		5.25	Y	100	590	685	775	3530	4920	7190	86	88	98	81	91	108

[a] Each value is average of three tests.

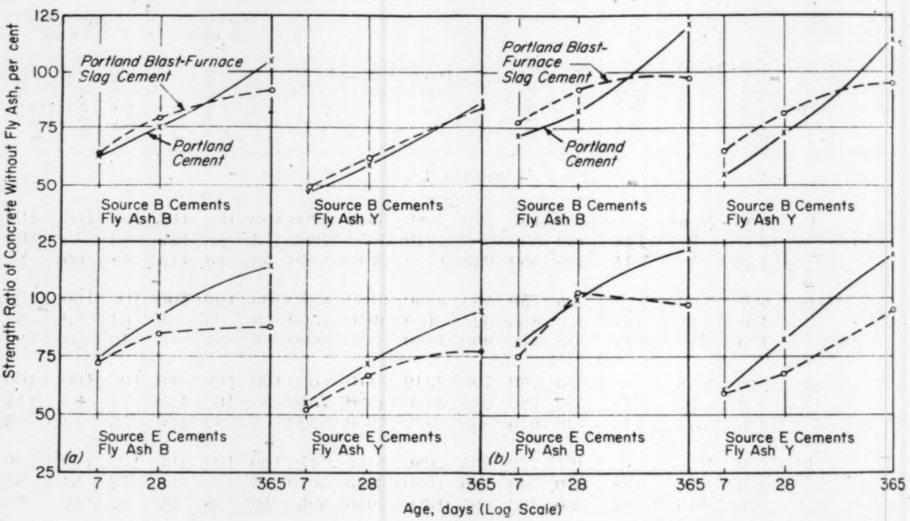
[b] Based on strength of concrete without fly ash. (Relative Strength in previous report.)

MIXING, FABRICATING AND CURING OF SPECIMENS

All mixing was done in an open-pan type mixer in accordance with usual laboratory procedures. The fly ash was added to the mix with the cement. All tests on the plastic and hardened concretes were made in accordance with the applicable ASTM method. The specimens prepared for strength tests were 6 by 12-in. cylinders and 6 by 6 by 21-in. beams which were tested at ages of 7 and 28 days, and 1 yr. All specimens were stored in moist air until tested.

Freezing-and-thawing tests were made on 3 by 4 by 16-in. beams in accordance with the ASTM Method C 292[6] for slow freezing and thawing in water. Freezing and thawing was continued for 250 cycles or until the specimens showed an average loss in N^2 of 40 per cent.

Outdoor exposure tests were made on 16 by 24 by 4-in. slabs. A dam was cast around the top perimeter of each specimen and when freezing and thawing was expected, the top surface was covered with ¼ to ½ in. of water. Each morning after the water had frozen, flake calcium chloride was spread over the ice at a rate of 2.4 lb per sq yd of surface. After the ice had melted, the chloride solution was washed off and fresh water left on the surface of each slab. The slabs were examined periodically and rated for amount and depth of scaling of the exposed surface. They were subjected to a total of 40 applications of calcium chloride.



(*a*) On compressive strength. (*b*) On flexural strength.

FIG. 1.—Effect of Replacement of One Third of Portland or Portland Blast-Furnace Slag Cement by Fly Ash.

STRENGTH TESTS

The results of the compressive[7] and flexural strength[8] tests on concrete with and without fly ash replacement are given in Table III. This table also gives the strength ratios of the concrete prepared with fly ash expressed as a percentage of the strength of the corresponding concrete without fly ash. This was referred to in the previous report as relative strength.

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[6] Tentative Method of Test for Resistance of Concrete Specimens to Slow Freezing and Thawing in Water or Brine (C 292 – 57 T), 1958 Book of ASTM Standards, Part 4.

[7] Method of Test for Compressive Strength of Molded Concrete Cylinders (C 39 – 59), 1958 Book of ASTM Standards, Part 4.

[8] Method of Test for Flexural Strength of Concrete (Using Single Beam with Third-Point Loading) (C 78 – 59), 1958 Book of ASTM Standards, Part 4.

Average values for the compressive strength ratios of the concrete for series I at various ages are shown in Fig. 1(*a*). A direct comparison is shown between the concretes prepared with the portland cement and the slag cement from the same source.

The upper diagrams of Fig. 1(*a*) show the compressive strength ratios of the concretes prepared with the two types of cement from sources B and containing the two fly ashes as replacements for $\frac{1}{3}$ of the cement. These diagrams show that at 7 and 28 days the strength ratios of the slag cement concretes were slightly higher than the strength ratios of the corresponding portland-cement concretes, regardless of whether fly ash B or fly ash Y was used as a replacement. At 1 yr, the strength ratios of the portland-cement concretes were higher than the strength ratios of the slag cement concretes for both fly ashes B and Y. Values given in Table III show that at 28 days and 1 yr, the actual compressive strengths of the concretes prepared with the slag cement from source B, with and without fly ash, were higher than the actual strengths of the corresponding concretes prepared with portland cement.

The lower diagrams of Fig. 1(*a*) show the compressive strength ratios of the concrete prepared with the two types of cement from source E, with the two fly ashes as replacements for $\frac{1}{3}$ of the cement. At all ages, the strength ratios of the concretes prepared with the portland cement were greater than the strength ratios of the portland blast furnace slag cement concretes. At 7 and 28 days, there was little difference between the strength ratios of the concrete prepared with the two types of cement, but at 1 yr the difference between them was pronounced. Table III shows that the actual compressive strengths at all ages were lower for the concrete prepared with the slag cement from source E than for the corresponding portland-cement concrete.

The compressive strength ratios of the concrete prepared with the portland blast furnace slag cement and containing $\frac{1}{3}$ fly ash replacement were lower than 100 per cent. In the four combinations of cement and fly ash, these ratios increased as the age of the concrete increased. It appears unlikely, however, that these strengths of fly ash concrete would eventually equal the strengths of the corresponding concrete without fly ash. Conversely, at 1 yr the compressive strengths of the concretes containing portland cement and fly ash B had exceeded the strengths of the corresponding concretes without fly ash.

The flexural strength ratios of the concretes for series I are shown in Fig. 1(*b*). The upper diagrams show the flexural strength ratios of the concretes prepared with the two fly ashes as replacements for $\frac{1}{3}$ of the two types of cement from source B, and the lower diagrams show the flexural strength ratios for concretes prepared with cements from source E.

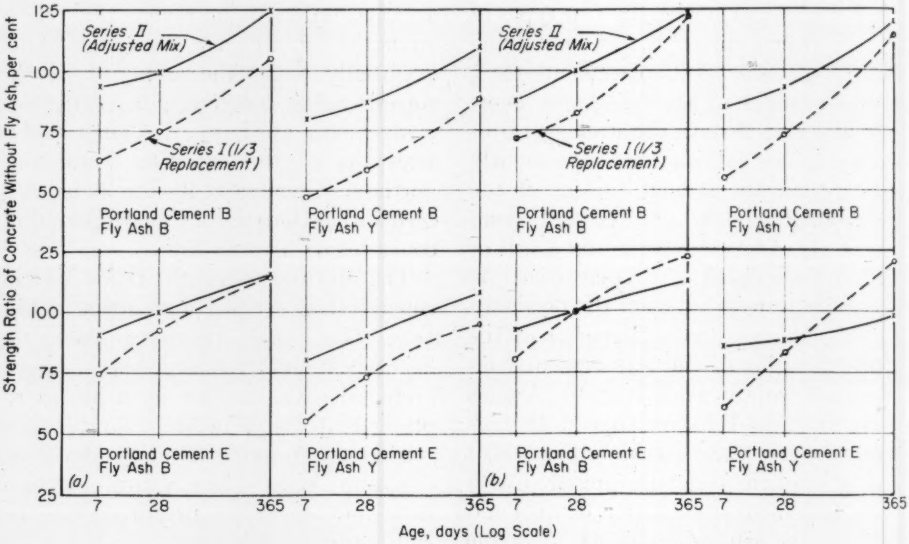
The same trends are shown for flexural strengths as were shown for compressive strengths. That is, the strength ratios of the concretes prepared with the portland blast furnace slag cement from source B, were higher at 7 and 28 days and lower at 1 yr than the strength ratios of corresponding concretes prepared with the portland cement. The strength ratios of the concretes prepared with the portland blast furnace slag cement from source E were lower at all ages, except for the 28-day test of concrete containing fly ash B. Table III shows that at 1 yr the actual flexural strengths of the portland blast-furnace slag cement concretes with $\frac{1}{3}$ fly ash were higher than the actual strengths of the concretes prepared with portland cement without fly ash.

Strength tests using the adjusted mix recommended by the producer of fly ash were made only with the type I portland cement. The results of these tests and the strength ratios of this concrete are given in Table III as series II and are shown in Fig. 2. This figure also shows a comparison between the strength ratios of the concretes for series II and similar concretes for series I.

In these tests, the effect on the strength of the concrete of both the source of the cement and of the fly ash is quite marked. Figure 2(*a*) shows higher compressive strength ratios at all ages for the adjusted mix of 5.25 bags of cement per cu yd than for mix with a cement content of 4.0 bags.

Figure 2(*b*) shows equal or higher flexural strength ratios at 7 and 28 days for the adjusted mix in all cases. At 1 yr the adjusted mix for the concretes prepared with cement B with both fly ashes had higher strengths, but when cement E was used, the strengths were lower.

The results of these tests support the claim of the fly ash producer that the adjusted mix will give good concrete strengths at early ages. The average compressive strength ratio of the adjusted mixes prepared with fly ash B at 28 days was 100 per cent and for fly ash Y it was approximately 90 per cent. Likewise, the flexural strength ratios averaged 100 per cent for fly ash B and 90 per cent for fly ash Y.



(*a*) On compressive strength. (*b*) On flexural strength.

FIG. 2.—Effect of Adjusted Fly Ash Mix.

LABORATORY FREEZING-AND-THAWING TESTS

Freezing-and-thawing tests were made in accordance with ASTM Method of Test for Resistance of Concrete Specimens to Slow Freezing and Thawing in Water or Brine (C 292).[5] Tests were made using all four cements and both percentage replacements of the two fly ashes. The results of these tests are given in Table IV. This table gives the loss at

50, 100, 150, 200, and 250 cycles and the durability factor, calculated on the basis of 250 cycles or a maximum loss in N^2 of 40 per cent. In addition, the relative durability of the concrete is given. This is expressed as the ratio of the durability factor of the concrete with fly ash replacement to that of the corresponding concrete without fly ash.

The relative durability factors are shown in Fig. 3. When fly ash B was used as a replacement, the relative durability in all cases was 90 per cent or more and in six of eight cases it was over 100 per cent. With this fly ash and a cement content of 4.0 or 5.25 bags, more durable concrete was usually obtained than with a 6.0-bag air-entraining concrete not containing fly ash. When fly ash Y was used, durable concrete was obtained only

TABLE IV.—RESULTS OF LABORATORY FREEZING-AND-THAWING TESTS.[a]

Cement			Fly Ash		Loss in N^2 at Cycles,[b] per cent					Durability Factor	Relative Durability,[c] per cent
Identification		Amount, bags per cu yd	Identification	Amount, lb per cu yd	50 cycles	100 cycles	150 cycles	200 cycles	250 cycles		
Source	Type										
SERIES I											
B.......	I	6.0	None	...	1	11	38	(159)		38	100
		4.0	B	148	0	6	31	(174)		42	111
		4.0	Y	144	1	14	28	(195)		47	124
B.......	IS	6.0	None	...	0	0	28	(195)		47	100
		4.0	B	148	0	0	7	32	(215)	52	111
		4.0	Y	144	5	18	(145)			35	74
E.......	I	6.0	None	...	0	0	3	11	20	80	100
		4.0	B	148	0	0	0	5	11	89	111
		4.0	Y	144	0	11	33	(166)		40	50
E.......	IS	6.0	None	...	0	0	9	40	(200)	48	100
		4.0	B	148	0	0	21	(178)		43	90
		4.0	Y	144	0	(85)				20	42
SERIES II											
B.......	I	6.0	None	...	1	11	38	(159)		38	100
		5.25	B	100	0	6	35	(166)		40	105
		5.25	Y	100	0	18	35	(166)		40	105
B.......	IS	6.0	None	...	0	0	28	(195)		47	100
		5.25	B	100	0	0	16	40	(200)	48	102
		5.25	Y	100	0	32	(108)			26	55
E.......	I	6.0	None	...	0	0	3	11	20	80	100
		5.25	B	100	0	0	1	23	28	72	90
		5.25	Y	100	0	1	12	38	(205)	49	61
E.......	IS	6.0	None	...	0	0	9	40	(200)	48	100
		5.25	B	100	0	0	5	17	30	70	146
		5.25	Y	100	0	22	(130)			31	65

[a] Each value is average of three tests.
[b] Figures in parentheses indicate number of cycles at which a loss of 40 per cent was reached.
[c] Based on durability of concrete without fly ash.

with portland cement from source B. With this high-carbon fly ash, the concretes prepared with portland blast-furnace slag cement from source B and both cements from source E showed poor durability.

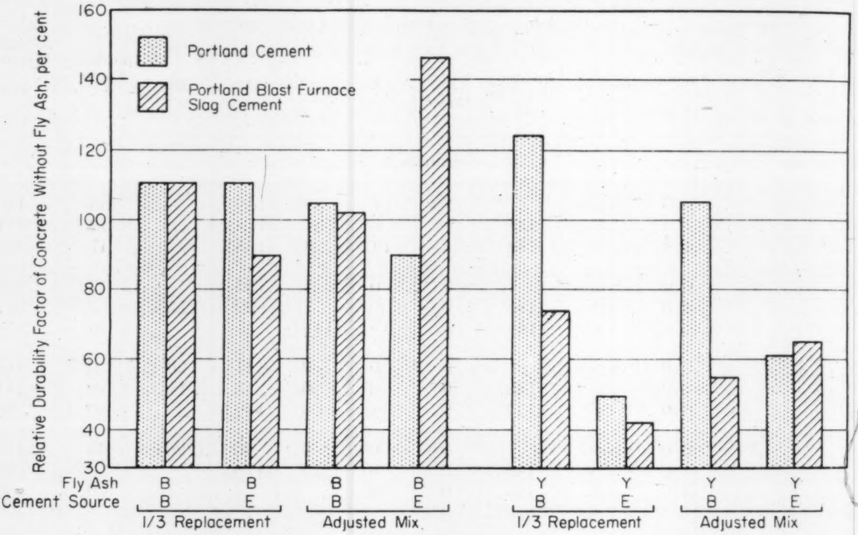


FIG. 3.—Effect of Fly Ash Replacement on Durability.

TABLE V.—RESULTS OF OUTDOOR SCALING TESTS.

Cement			Fly Ash		Air, per cent	Rating Freezing and Thawing with CaCl₂	
Identification		Amount, bags per cu yd	Identification	Amount, lb per cu yd		15 cycles	40 cycles
Source	Type						
B............	I	6.0	None	None	5.3	2½	4½
		5.25	B	100	4.5	3	4½
		5.25	Y	100	4.5	4½	7
E............	I	6.0	None	None	4.8	2	4
		5.25	B	100	6.2	2	3
		5.25	Y	100	5.3	5	7½

When all mixes containing fly ash were considered, it was found that those prepared with portland cement had a somewhat greater relative durability (95 per cent average) than those prepared with portland blast-furnace slag cement (86 per cent). Comparisons between the fly ash concretes of series I (4.0 bags of cement) and those of series II (5.25 bags of cement) showed both to have very nearly the same relative durability. However, the portland blast-furnace slag cements of series I had a lower durability factor (79 per cent) than those of series II (92 per cent).

These tests indicate that satisfactory

durability was obtained on concrete when fly ash B (low-carbon fly ash) was used. This applied to all sources and types of cement used. The tests also indicated that poor durability was usually obtained for concrete when fly ash Y (high-carbon fly ash) was used. Only the concrete prepared with the portland cement from source B gave satisfactory durability with this fly ash.

OUTDOOR SCALING TESTS

Table V shows the results of the periodic examinations of the slabs which were exposed to outdoor freezing and subsequent thawing with calcium chloride. Tests were made on concretes prepared with the portland cements only, and when fly ash was used as a replacement of cement, the amount was that recommended by the producer. The specimens were rated according to the amount and depth of scaling. A rating of 0 indicated no scaling and a rating of 10 indicated deep scaling over the entire surface of the specimen.

These tests showed that at 40 cycles the concrete which contained fly ash B had resistance to scaling equal to or slightly better than similar concrete without fly ash. Where fly ash Y was used, however the resistance was poorer than that of the concrete without fly ash.

CONCLUSIONS

These two series of tests verify some of the conclusions of the previous report. Mainly, that the properties of the concrete are affected by the carbon content of the fly ash used as a replacement for part of the cement. Lower strengths and durability were obtained with concrete containing the high-carbon fly ash than when the low-carbon fly ash was used.

Fly ash of low carbon content can be used as a replacement for a part of the portland blast-furnace slag cement without serious decrease in the strength or durability of the concrete.

Concrete prepared with low-carbon fly ash as a replacement for portland cement in the amounts recommended by the producers of fly ash, has compressive and flexural strengths at 28 days equal to similar concrete without fly ash.

The recommended amount of fly ash, if low in carbon content, will also produce concrete that will have resistance to scaling caused by the use of deicing agents equal to that of similar concrete without fly ash.

Tests for strength and durability of concrete indicate that, for optimum effectiveness, the amount of fly ash used should be correlated with the amount of lime released by the portland cement. If sufficient lime is not available to react with all of the fly ash, some of the latter is only an inert filler and will tend to impair the quality of the concrete.

It is suggested that the amount of fly ash used should be about 15 per cent by weight of the CaO content of the cement as determined by chemical analysis. This amount may vary somewhat depending upon the chemical composition of the aggregates.

DISCUSSION

Mr. Paul Klieger.[1]—Do you have any deicer scaling tests of concretes, both nonair-entrained and air-entrained, made with portland blast-furnace slag cements and fly ash replacements? Recent tests in our laboratories indicate that fly ash replacements are detrimental to resistance to deicer scaling, despite the maintenance of air content. This was more marked in air-entrained concretes made with type IS cements than for similar concretes made with a blend of four type I cements.

Mr. W. E. Grieb (*author*).—No scaling tests were made with portland blast-furnace slag cement.

Mr. J. J. White.[2]—The speaker has referred to the presence of free carbon in fly ash. It seems to me that undue stress is laid upon the permissive amount of free carbon in fly ash intended for use in combination with portland cement. ASTM Specification C 350 - 60 T[3] sets the maximum limit for loss on ignition at 12 per cent. Some authorities will not permit more than 6 per cent. It is my understanding that the most serious effect of a high percentage of fly ash is the increased demand for mixing water when used with portland cement, and, under the influence of certain combinations of pozzolanic activity and particle fineness, can yield poor results. Perhaps more attention should be paid to the pozzolanic activity index rather than loss on ignition. It is my understanding that fly ash with relatively high carbon content and high pozzolanic activity index has often shown far better results when used in portland cement concrete than fly ash low in both instances.

When evaluating the suitability of fly ash for use in combination with portland cement, whether in soil cement, mortar or concrete, three factors must be considered, that is, carbon content, pozzolanic activity and fineness. Fly ash with high carbon content, high pozzolanic activity and low fineness can yield good results; high carbon content, low pozzolanic activity and high fineness may be expected to yield poor results and low carbon content, high pozzolanic activity and high fineness may be expected to yield excellent results. The consideration of any one, alone, of the three factors is certainly not conclusive.

Mr. W. E. Grieb (*authors' closure*).—We agree with Mr. White that the pozzolanic activity index should also be considered when evaluating the suitability of fly ash for use in concrete. However for these tests, the carbon content was an excellent indication of the durability of the concrete.

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[1] Portland Cement Assn., Chicago, Ill.

[2] Parsons, Brinckerhoff, Quade & Douglas, Engineers, New York, N. Y.

[3] Tentative Specification for Fly Ash for Use as an Admixture in Portland Cement Concrete (C 350 - 60 T), 1960 Supplement to Book of ASTM Standards, Part 4.

INVESTIGATION OF CONCRETE MATERIALS FOR THE SOUTH SASKATCHEWAN RIVER DAM*

By G. C. Price[1]

Synopsis

Concrete requirements resulted in a modified specification for type V cement, the possible use of pozzolan, and the upgrading of local aggregates. Studies of concrete and mortar were conducted to evaluate sulfate resistance, cement-aggregate reaction, compressive strength, and mix characteristics. Seven type V cements and one type I cement were used with fly ash and pumicite. Mixtures were designed on the basis of fixed cement factor with increments of pozzolan replacing a portion of the aggregate. The program is in a short-term stage with observations ranging up to twelve months. Trends in performance of concrete, the mortar phase of concrete, and standard sand mortars in mixed sulfate solution are described. A low percentage of reactive siliceous shalestone in the processed fine aggregate produced no deleterious effect in concretes and mortars stored in humid air. The effect of pozzolans upon the mixtures is described, and compressive strengths are given for ages up to 1 yr. Long-term observations of exposed concretes in sulfate soils have revealed little deterioration where proper techniques in design, manufacture, and placement result in a highly impermeable concrete.

Structures appurtenant to a large earth dam in central Saskatchewan (South Saskatchewan River Dam) will require the placement of 500,000 cu yd of concrete. About one half of this amount will be used in the construction of five 20-ft diameter tunnels for river diversion and power development. The tunnels will be driven through a hard zone of the Bearpaw shale which, although classed geologically as a shale, is regarded by many engineers as a hard clay. Portions of the tunnel, stilling basin, spillway, and irrigation outlet structures will be constructed adjacent to Bearpaw shale, glacial clay, or alluvial clay. Concrete slabs and walls in these structures will be separated from the soil by granular material. Wall footings and cutoffs will be directly in contact with the soil. Sulfate concentrations at the structure sites are high, ranging from 0.2 to 1.1 per cent in the soil and 1000 to 8000 ppm in the ground water.

Climatic, material, and foundation conditions at the dam combine to present problems in development of a concrete that will resist sulfate attack, weathering, alkali-aggregate reaction, early loading, and abrasion.

A study of these problems resulted in the careful upgrading of local granular materials—in particular, the heavy-media processing of coarse aggregate to

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[1] Concrete Engineer, Prairie Farm Rehabilitation Administration, Canada Department of Agriculture, Saskatoon, Sask., Canada.

remove deleterious lightweight materials. Siliceous shalestone, a predominant component of the lightweight fraction, was found to be reactive with alkalies in cement. While almost all of this was removed from the coarse aggregate, a small percentage remaining in the processed fine aggregate presented the possibility of a harmful reaction occurring in concrete.

The study also pointed to the need for a cementitious material that would: (1) effectively resist attack from strong sulfate concentration in soils and ground water at the site, (2) provide optimum workability in concrete for the difficult placing of the thin, heavily reinforced tunnel lining, and (3) provide adequate strength-gain in construction of tunnel linings where early loading is expected and for added protection from frost damage during winter construction of spillway and irrigation outlet structures.

With these factors in mind, a research program was undertaken to investigate concrete and mortars containing a number of sulfate-resistant cements—alone and in combination with pozzolans. The tests, which centered largely around long-term studies of sulfate resistance, were begun in March, 1959.

The rather large research program involves 200 concrete mixes and 300 mortar mixes. The performance of 5000 mortar and concrete specimens is being observed in various storage conditions: sulfate solution, air at 100 F and 100 per cent relative humidity, air at 72 F and 100 per cent relative humidity, and natural exposure in Bearpaw shale at the dam site.

In a rather unusual approach to the use of pozzolans, aggregate was replaced rather than cement. This paper describes some of the methods used and covers short-term results which in some cases have shown interesting trends. No attempt is made to present a complete picture or draw definite conclusions, in view of the very preliminary nature of the studies, it is expected that this will be done in about 5 yr, when long-term results will be available.

AGGREGATE SOURCE AND PRELIMINARY STUDIES

In the early stage of the investigation, a promising source of aggregate was found in a flat-topped glacial outwash deposit strategically situated on the west abutment of the dam. This potential source was named the plateau deposit. Exploration and testing of the plateau granular materials between 1950 and 1958 revealed some shortcomings with respect to gradation and quality and pointed to the need for upgrading the materials. As a result, the first aggregate production contract called for thorough processing to remove clay sizes and coatings, close sizing to assist in quality control, and heavy-media processing of the coarse aggregate to remove deleterious lightweight material.

The predominant harmful lightweight material was siliceous shalestone—an absorptive rock with low resistance to weathering. Deleterious heavyweight material, which could not be removed by the heavy-media process, was also present—as for example the greater portion of the ironstone constituent.

Some consideration was given to removing at least a portion of the lightweight deleterious materials in the fine aggregate by applying the heavy-media process to the No. 4 to No. 8 size fraction. While it was expected that this could be done successfully, it was not required since it was doubtful if the added cost would be justified.

In 1959 the use of ASTM Method C 227[2] revealed that the natural pit-run sand from the plateau deposit was potentially reactive with the alkalies in cement. The main reactive material as determined through petrographic examination of mortar, concrete, and aggregates was the siliceous shalestone which was estimated to contain about 80 per cent opal. All of this material was removed from the coarse aggregate by the heavy-media process. In the processed fine aggregate, shalestone content ranged from 0.6 to 1.3 per cent by weight, averaging 0.8 per cent. The average amount of reactive opal occurring in a typical concrete mixture using the processed aggregates was estimated to be 0.3 per cent by weight.

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[2] **ASTM Method of Test for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar Bar Method), (Tentative) (C 227 - 60 T), 1960 Supplement to ASTM Book of Standards, Part 4.**

A survey of 45 existing structures and a number of specimens from a natural exposure site at the dam provided evidence that concrete made with the relatively low-alkali western cements and containing aggregates from the plateau deposit and many other sources in Saskatchewan apparently did not suffer appreciable damage from alkali-aggregate reaction even over an extended period in a damp environment. It also indicated that the potential danger of reaction was a real one, as revealed by the presence of slightly reacted particles of siliceous shalestone in many of the specimens examined.

STUDIES OF CONCRETE AND MORTAR CONTAINING CEMENT-POZZOLAN COMBINATIONS

A decision to use sulfate-resistant cement was made in the early planning of the project, and the cooperation of the cement companies was enlisted to obtain a modified product having the early strength properties of present-day normal portland cements. The use of pozzolan as a replacement for aggregate was considered for the high workability requirement in tunnel construction and possibly for improved sulfate resistance over that obtainable with sulfate-resistant cement alone. The discovery that certain constituents in the fine aggregate from the dam site were reactive with alkalies in cement suggested the use of cement with total alkali content below 0.6 per cent and placed additional emphasis on the use of pozzolan as an inhibitor of the alkali-aggregate reaction.

The evaluation of sulfate resistance as affected by the combinations of pozzolan and type V cement in concrete was a relatively new territory. One comprehensive study at the University of California (**1**)[3] had concluded that the replacement of type V cement with pozzolan produces no improvement in sulfate resistance.

On the majority of jobs where pozzolan is used as a replacement for cement, the amount of cement in the mix is low—on the order of 3 to 4 sacks per cu yd. While this is desirable in many forms of mass concrete, it might be an unsatisfactory approach in structural concrete that will be subjected to early loading, sulfate attack, and abrasion, all within a relatively short time after construction.

Experts in the field of sulfate resistance have stressed the importance of adequate cement content (**2–4**). For the appurtenant structures at the dam, it seemed advisable to choose a minimum cement factor and use pozzolan as a replacement for aggregate rather than cement.

Lean, medium, and rich concretes of similar workability were designed in which varying increments of pozzolan were added to constant cement factors and compensated for by adjustments in aggregate and water content. Concretes were made with processed aggregates from the dam site, a number of standard and modified type V cements, a type I cement, and two pozzolans. A study of lean and medium, standard sand mortars of constant flow with varying additions of pozzolan was also made.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

Particular emphasis was placed on the sulfate resistance of concrete and mortar as measured by long-term length and weight changes of specimens immersed in a magnesium-sodium sulfate solution. Individual containers were used for each set of specimens in sulfate solution or distilled water. The solutions were changed each time the specimens were measured—at intervals of approximately two months. All containers were stored in a temperature-controlled room at 71 ± 2 F. Tests were also made to evaluate the problem of alkali-aggregate reaction; this entailed the length change of mortar and concrete specimens in humid storage of 100 F and 72 F, respectively.

Cements and Pozzolans:

One type I and seven type V cements covering a fairly wide range of chemical and physical make-up were used in the tests. A number of the type V cements, which were all produced in western Canada, were manufactured especially for the investigation. Test data for the cement are given in Table I.

TABLE I.—PROPERTIES OF CEMENTITIOUS MATERIALS.

PROPERTIES OF CEMENTS

Cement Designation	Cement Type	Hypothetical Compound Compositon, per cent by weight					Oxide Composition, per cent by weight			Blaine Fineness, sq cm per g
		C_3S	C_2S	C_3A	C_4AF	$CaSO_4$	Total Alkalies as Na_2O	MgO	Free CaO	
A	V	43.8	33.8	2.0	13.8	2.75	0.42	2.64	0.24	3039
B	V	57.5	19.4	0.8	12.8	4.7	0.27	2.68	0.04	4744
C	V	43.3	35.5	2.1	12.0	2.7	0.54	1.40	0.34	2768
D	V	44.7	32.5	4.0	10.8	3.1	0.56	1.42	0.48	2872
E	V	43.7	32.8	2.8	12.8	2.8	0.55	1.40	1.37	3153
F	V	47.5	31.1	0.4	13.5	3.0	0.37	2.48	0.32	3329
G	V	45.9	30.3	2.6	12.7	3.0	0.58	1.44	1.17	3121
H	I	47.1	24.6	10.0	7.9	4.3	0.96	3.43	0.69	3214

PROPERTIES OF POZZOLANS

Pozzolan Designation	Ignition Loss, 800 C, per cent by weight	Oxide Composition, per cent by weight				Blaine Fineness, sq cm per g	Retained on No. 325 Sieve, per cent by weight	Pozzolanic Activity, psi	Specific Gravity
		$SiO_2 + Al_2O_3 + Fe_2O_3$	MgO	SO_3	Available Alkali as Na_2O				
Fly ash I*a*	0.21	70	4.43	0.42	1.33	2070	19.6	647	2.44
Fly ash I*b*	0.31	67	4.24	0.61	3.10	1960	21.7	563	2.45
Pumicite II	3.56	87	1.23	0.12	0.84	6875	3.0	669	2.50

In the investigation, emphasis was placed on a fly ash because of the strategic location of the material with respect to the project. The two shipments of fly ash used in the tests were given separate designations because of some slight differences in chemical and physical characteristics. One natural pozzolan—pumicite—was used for comparative purposes. Test data for the pozzolans are also given in Table I.

Aggregates:

Processed plateau-deposit aggregates sampled from stockpiles at the dam site were used in all phases of the concrete tests. The aggregates were batched to the test mixes in four sizes: 1½ to ¾ in., ¾ to ⅜ in., ⅜ in. to No. 4, and fine aggregate passing No. 4.

Concrete Mixes:

With the aggregates as processed and using a water-cement ratio of 0.50 by weight for the tunnel concrete, the cement factor would normally be on the order of 5½ Canadian sacks (87.5 lb per sack) per cu yd. A reasonable minimum appeared to be 5 sacks per cu yd with the pozzolan addition equal to or greater than the ½ sack reduction from the normal cement factor in terms of solid volume. This formed the basis for the first phase of testing in which varying increments of pozzolan were added to a constant 5-sack cement factor. In the second phase of the program, minimum cement factors of 4 and 6 sacks were also used. Essentially, therefore, the tests involved the addition of varying increments of pozzolan to constant cement factors in rich, medium, and lean concretes and comparison of these with straight cement concretes.

TABLE II.—COMPARATIVE CONCRETE MIX SERIES.
Sacks of Cement + Pozzolan by Absolute Volume.

Series	Cement Addition	Reference Mix	Pozzolan Additions		
I	5 + 0	4 + 0	4 + 1	4 + 1¾	4 + 2½
II	6 + 0	5 + 0	5 + 1	5 + 1¾	5 + 2½
III	7 + 0	6 + 0	6 + 1	6 + 1¾	6 + 2½

For convenience, the additions were made in terms of sacks per cu yd, each sack of pozzolan being equal by absolute volume to one sack of cement. Throughout the tests, additions comprised 1, 1¾, and 2½ sacks of pozzolan. The specific gravity of the pozzolan was lower than that of cement; therefore 1 sack of pozzolan weighed considerably less than 1 sack of cement. For example, the weight of fly ash I to give an equivalent absolute volume of one 87.5-lb (Canadian) sack of cement is 68 lb. By weight, the above additions of fly ash would be approximately 0.8, 1.4, and 1.9 sacks, respectively. The comparative mix series used are summarized in Table II.

The decision was made to use entrained air in the tunnel concrete to obtain superior placing characteristics and sulfate resistance (2,5). Air-entrained concrete was accordingly used throughout the testing program.

TABLE III.—RANGE OF CONSISTENCY AND AIR CONTENT.

	Range in Slump, in.	Range in Air Content, per cent by volume
Phase I	3 to 4	4 to 5
Phase II	3.5 to 4.5	4.2 to 5.5

The mixes were designed on the basis of absolute volume to meet a range of consistency and air content shown in Table III. Slump and air content were increased in phase II to obtain an improved mixing action and to approach conditions more like those expected in the tunnel construction.

The size of the test batch was increased from 220 lb in phase I to 350 lb in phase II so that alkali-aggregate test beams, in addition to sulfate-resistance and compressive-strength specimens, could be molded from the same mix.

An accurate estimate had to be made of unit water requirement for the mix to meet the specified cement factor, pozzolan addition, range of consistency, and air content. This was complicated by the addition of pozzolan, which resulted in a decreased ratio of fine to coarse aggregate owing to improved workability and increased, or decreased, water requirement depending upon the characteristics of the pozzolan. As a result, there were a number of "waste" mixes which failed to meet the requirements for slump and air content. The number of such waste mixes lessened as the laboratory staff became familiar with the characteristics of each pozzolan. Very few repeat mixes were required with the fly ash, as it had stable mix characteristics. On the other hand, a marked increase in requirements for water and air-entraining agent resulting from additions of the pumicite gave difficulty in designing to meet the mix specifications.

Mortar Mixes:

Tests involving the addition of pozzolan to constant cement factors in mortar were designed to parallel the concrete program described previously. The mortar mixes were made with graded standard Ottawa sand for an evaluation of compressive strength and sulfate resistance and with processed fine aggregate for studies of alkali-aggregate reactivity.

The mortar proportions for the control mixes containing no pozzolan were determined on the basis of two selected water-cement ratio combinations:

1. Water-cement ratios of 0.50 and 0.60 (medium mortar), and

2. Water-cement ratios of 0.60 and 0.75 (lean mortar).

Proportions of cement and graded standard Ottawa sand to achieve the selected water-cement ratios in control mixes without pozzolan were first determined by testing mortar batches with flow in the range of 108 to 112. Density and yield of the plastic mortar were determined, and from these, cubic-yard weights of cement, sand, and water were calculated.

The cement content of the high water-cement ratio control mortar became the constant factor in subsequent mixes with pozzolan additions. The amount of pozzolan added to the first test mix was equal (in solid volume) to the difference in cement content of the low and high water-cement control mortars. Pozzolan additions to the constant cement content in the second and third test mixes were $1\frac{3}{4}$ and $2\frac{1}{2}$ times that used in the first test mix, respectively.

Cement content was maintained constant as nearly as practically possible by substituting pozzolan for sand on the basis of solid volume and at the same time maintaining the sum of absolute volumes of cement, pozzolan, and sand constant and equal to the amount of cement and sand in the high water-cement ratio control mortar.

Actually, the cement content did not remain exactly constant in the test mixes because of increased or decreased water requirement (for specified flow of 108 to 112) depending upon the characteristics and quantity of the pozzolan. In the concrete tests, this was overcome by redesigning and repeating mixes which could be adjusted generally in one retrial. Such a procedure was thought to be impractical for the mortar tests.

Nine 2-in. cubes and six 1 by 1 by $6\frac{1}{4}$-in. bars were molded from the Ottawa sand mixes for compressive-strength and sulfate-resistance studies, respectively. Four 1 by 1 by $11\frac{1}{4}$-in. bars were molded from the plateau fine aggregate mixes for alkali-aggregate studies.

A modification of the Canadian Standards Assn. (CSA) lean mortar bar method for determining sulfate resistance of cement[4] was used to evaluate the effect of substitutions of pozzolan for cement and aggregate.

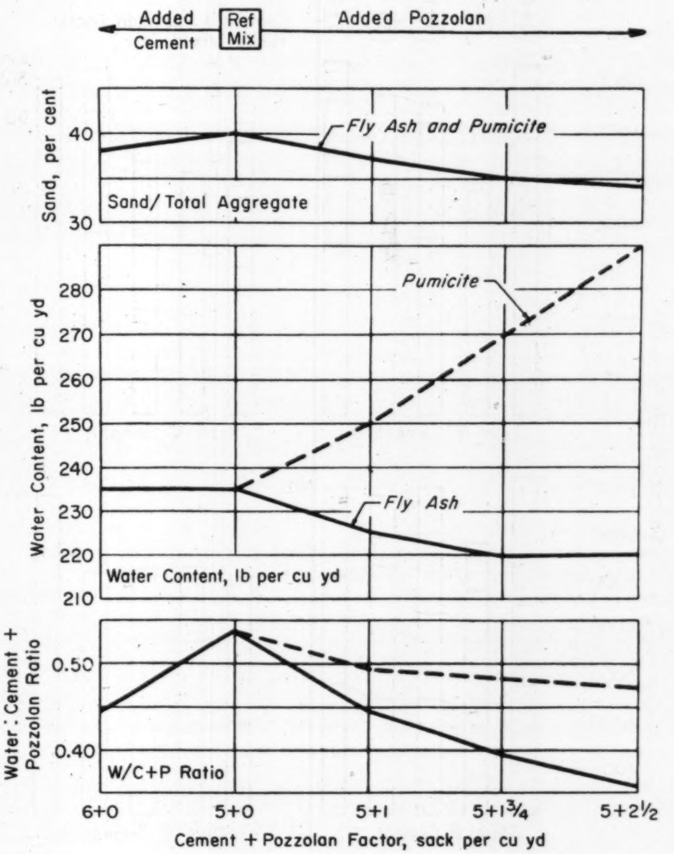

FIG. 1.—Concrete Mix Adjustment Necessitated by Pozzolan Additions.

In the standard CSA procedure, proportions of cement and standard Ottawa sand are 1:5 by weight. The percentage of water used in the mortar is based on the normal consistency and calculated from an empirical formula.[5] The resulting stiff consistency of the mortar is partly instrumental in producing a permeable material that allows diffusion of sulfate solution, thus exposing cementing material throughout the specimen to chemical action.

In modifying the procedure for replacement of cement and sand with pozzolan, an attempt was made to maintain the stiff consistency of the standard test. Since specific gravities of the pozzolans were different from the cement and sand, replacements were made on the basis of absolute volume in both the normal consistency and sulfate resistance procedures. Proportions for the test mixes were as follows:

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[4] "Specifications for Portland Cements," Canadian Standards Assn.—A5, 1961.

[5] ASTM Standard Method of Test for Tensile Strength of Hydraulic Cement Mortars, (C 190 – 59), 1959 Supplement to ASTM Book of Standards, Part 4.

Replacement of cement:

Cement + Pozzolan: Sand

= 1:6 by absolute volume

Replacement of sand:

Cement: Pozzolan + Sand

= 1:6 by absolute volume

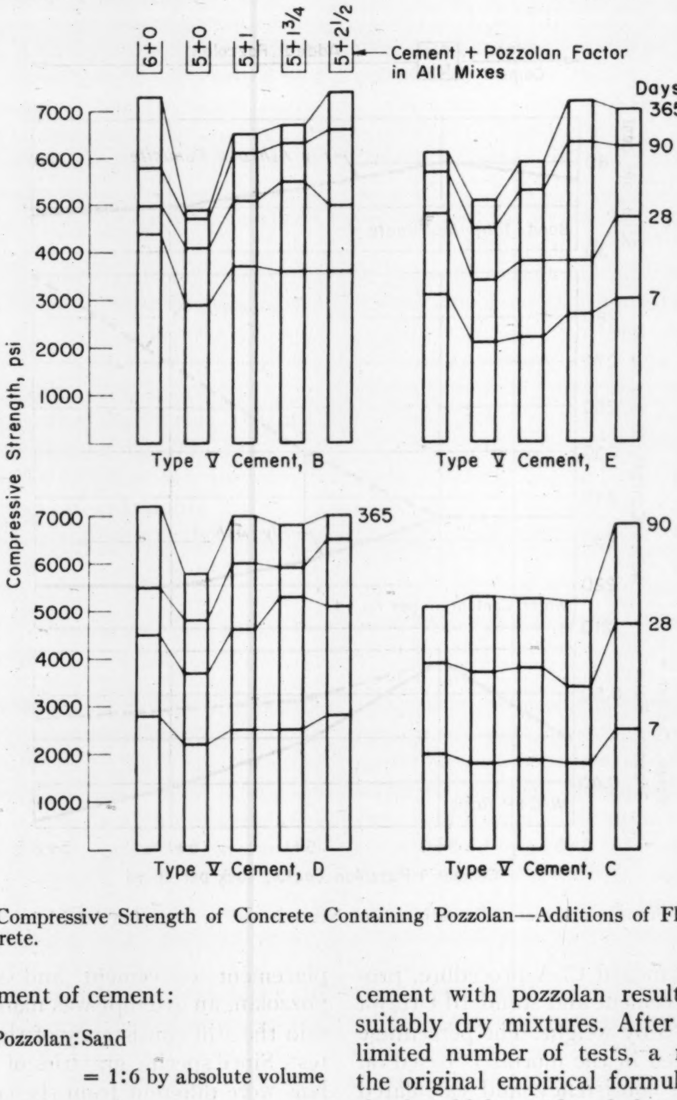

FIG. 2.—Compressive Strength of Concrete Containing Pozzolan—Additions of Fly Ash I*a* to 5-Sack Concrete.

In the tests replacing sand with pozzolan, it was found that the original method for determining batch weight of water produced the desired consistency. On the other hand, replacements of cement with pozzolan resulted in unsuitably dry mixtures. After running a limited number of tests, a revision of the original empirical formula was derived, which produced batch weights of water giving the desired stiff consistency.

Six 1 by 1 by $6\frac{1}{4}$-in. bar specimens fitted with metal reference points were molded from the lean mortar mixes for sulfate-resistance studies.

ASTM Method C 227[2] was also used to check the suitability of plateau fine aggregate in combination with high- and low-alkali cements. This involved hand mixing of 1:2.25 mortars with flow maintained between 105 and 120. Four 1 by 1 by 11¼-in. bars were molded for the alkali-aggregate studies.

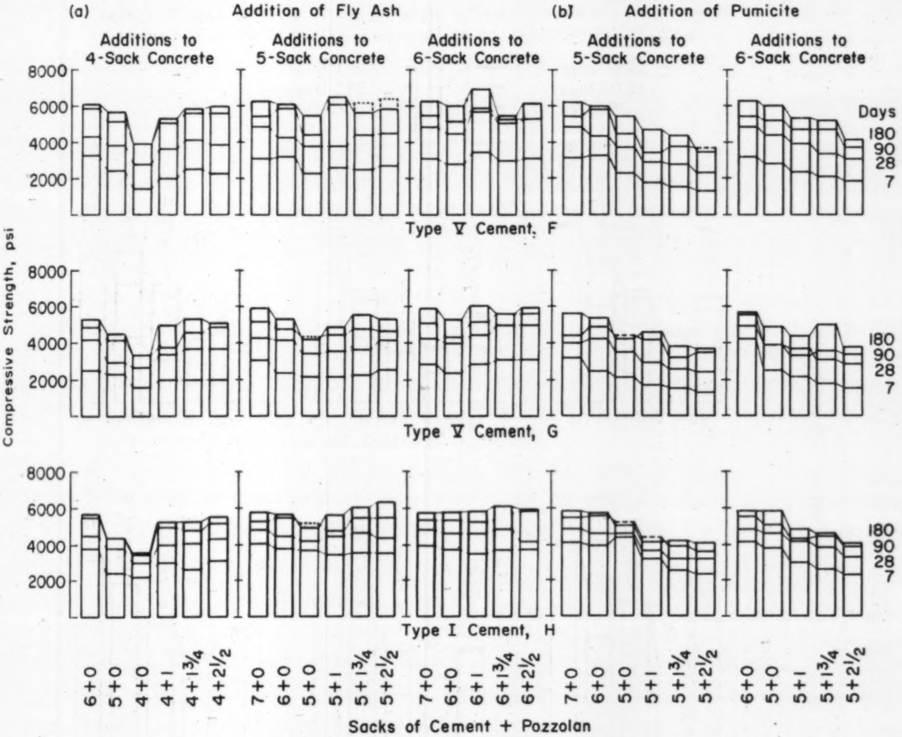

FIG. 3.—Compressive Strength of Concrete Containing Pozzolan.
(*a*) Additions of fly ash I*b* to 4, 5, and 6-sack concrete.
(*b*) Additions of pumicite II to 5 and 6-sack concrete.

Performance of Pozzolans in Plastic Concrete:

With increasing additions of the fly ash to fixed cement factors in concrete, water requirement was not increased, resulting in a lower ratio of water to total cementitious material. Workability and cohesiveness of the mix were improved, making possible an appreciable decrease in slump and a lower ratio of fine aggregate to total aggregate for a given placing condition. Requirement for air-entraining agent was slightly increased only with the largest additions of fly ash. Although some improvement in workability was obtained with additions of pumicite, water requirement was increased as much as 35 per cent to achieve the specified slump, and it was necessary to double the amount of air-entraining agent to obtain the desired air content. Mix adjustments necessitated by the pozzolan additions are illustrated in Fig. 1.

Compressive Strength of Concrete and Mortar:

The compressive strengths at various ages of concrete containing additions of pozzolans to constant cement factors are shown in Figs. 2 and 3. Specimens for compressive strength tests were cured in the fog room at 72 ± 1 F.

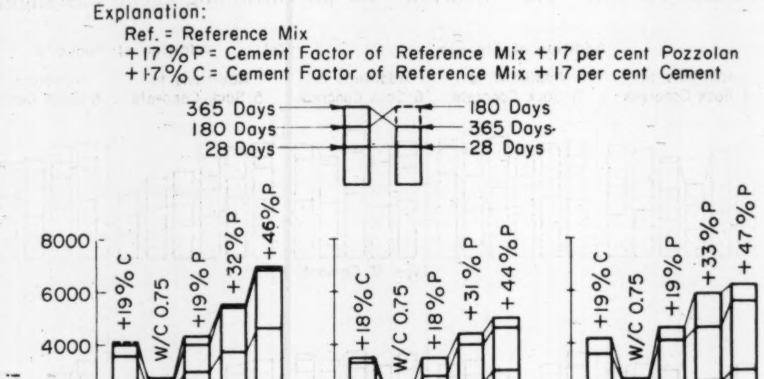

FIG. 4.—Compressive Strength of Mortar Containing Pozzolan—Additions of Fly Ash I*a* to Medium (*W*/*C* 0.60) and Lean (*W*/*C* 0.75) Mortars.

Addition of fly ash generally produced increased strength in concrete at all ages. The improvements were small at 7 days but ranged up to 1000 psi at 3 months and 1 yr. In a number of cases, a 1-sack addition of the pozzolan produced improvements that were equal to or greater than those attained by 1-sack addition of cement. The results suggested a satisfactory pozzolanic effect of the fly ash with commercial type V and type I cements. In contrast to the results with fly ash, the addition of pumicite resulted in reduced strengths at all ages.

The beneficial effect of fly ash on compressive strength was also illustrated in the tests with graded standard Ottawa sand mortars. Results of the addition tests involving combinations of fly ash Ia with three type V cements are shown in Fig. 4. Compressive strengths at various ages illustrate the relative effect of additions of cement and fly ash to fixed cement factors in medium and lean mortars. Three increments of fly ash were used in the comparison mixes; only one addition of cement was used—equal to the smallest increment of fly ash. The addition of 16 to 19 per cent fly ash resulted in increased strengths over the reference mixes at all ages, with 28-day strengths slightly less and 6-month and 1-yr strengths slightly greater than those attained by equal additions of cement. The addition of 32 to 47 per cent fly ash produced more substantial increases at all age levels—in every case greater than those resulting from the 16 to 19 per cent added cement. In the medium mortar series ($W/C = 0.60$), the increased strengths resulting from 27 to 44 per cent added fly ash ranged from 2000 psi at 28 days to 4000 psi at 1 yr. The standard sand mortar mixes were not performed with the pumicite.

Sulfate Resistance of Concrete and the Mortar Phase of Concrete:

To obtain some idea of how concrete might perform in the field, observations were made of concrete immersed in strong sulfate solution. It was realized that such an accelerated test would leave much to be desired, owing to the unnatural conditions of total immersion and high ratio of exposed surface to total mass. At best, it was felt that the difference obtained among various concretes stored under very adverse conditions in the laboratory would also occur under actual conditions in practice, but to a lesser degree.

Nine 3 by 6-in. concrete cylinders were molded from each test mix for the exposure tests. After a 28-day curing period, three were placed in distilled water, three in a mixed sulfate solution of $0.15M$ Na_2SO_4 + $0.15M$ $MgSO_4$, and three in natural Bearpaw shale at the dam site. Performance of the specimens was judged on the basis of weight change and visual observation, methods which had been used with success by various research workers (**1**,**8**). It is to be noted that performance of the specimens was judged on the basis of exposure at rest in the solution. No mechanical means was employed to remove loosened grain or flakes.

A proper evaluation of concretes made with type V cements would require a very long time. In an attempt to obtain early indications, the mortar phase of the various concretes under test was also compared by screening a portion of concrete from each mix over a No. 4 sieve to remove coarse aggregate sizes. The mortar thus obtained was molded into 1 by 1 by 6¼-in. bars fitted with metal reference points. After a 28-day curing period, the bars were immersed in distilled water and sulfate solution and length-change measurements recorded at intervals of two months.

Midway in the program, the procedure was modified in another attempt to obtain earlier indications of differences in potential sulfate resistance of concrete. Extra sets of 3 by 6-in. concrete cylinders and 1 by 1 by 6¼-in. mortar-phase bars were cast for storage in a stronger sulfate solution composed of 5 per cent Na_2SO_4 + 5 per cent $MgSO_4$. As before, performance of the concrete was judged on the basis of weight change and visual observation, and mortar on the basis of length change.

Test results for the sulfate-resistance studies of concrete are available for exposure periods ranging from 6 to 12 months. It is apparent that a much longer time will be required to evaluate the relative performance of materials.

The majority of concrete cylinders in 0.15*M* mixed sulfate solution gained in weight for 9 to 12 months, at which time a leveling out or a slight drop in weight occurred. In the initial phase with fly ash I*a* in combination with four type V cements, leveling out in weight gain occurred at 9 to 12 months, but there was no significant drop in weight after 1 yr exposure. This is illustrated in Fig. 5.

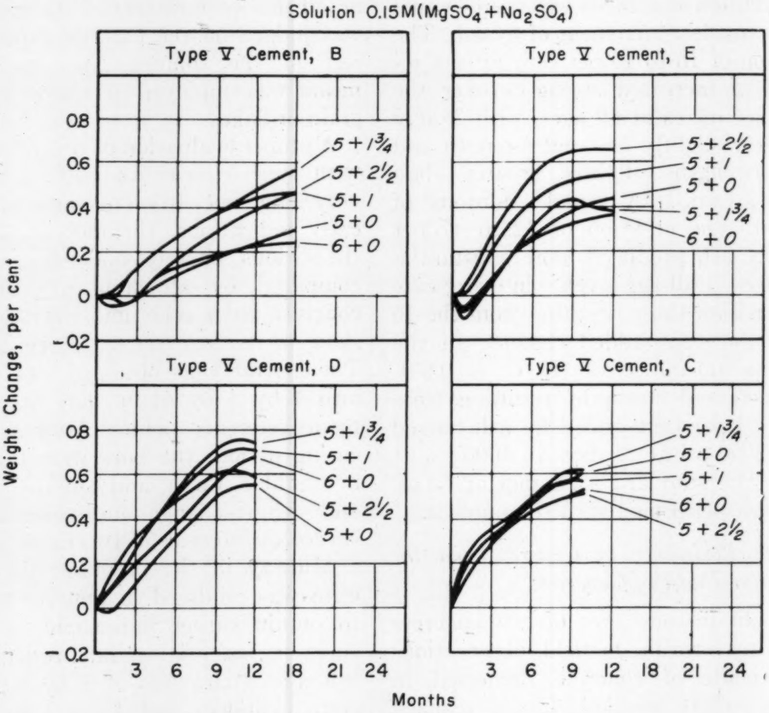

FIG. 5.—Weight Change of Concrete in 0.15*M* Mixed Sulfate Solution—Addition of Fly Ash I*a* to 5-Sack Concrete.

The situation was different for concrete cylinders in the more concentrated 5 per cent sulfate solution. In the second phase of testing, some 4 and 5-sack concretes containing additions of fly ash I*b* and pumicite II started to level off or lose weight at 5 months—more so than straight cement concrete. The loss was greatest for 4 + 2½-sack concrete containing type V cement F and fly ash I*b*, with test cylinders in the solution returning to their original weight at 9 months' exposure. Weight-change results for phase II are shown in Fig. 6.

Because of the rather inconclusive results revealed by weight change, an effort was made to rate performance on the basis of appearance of concrete in sulfate solution. Specimens were examined and photographed at 6-month intervals. The examination revealed that sulfate attack started in small surface pockets penetrating radially to weaken and destroy a thin surface film surrounding the void. This resulted in a charac-

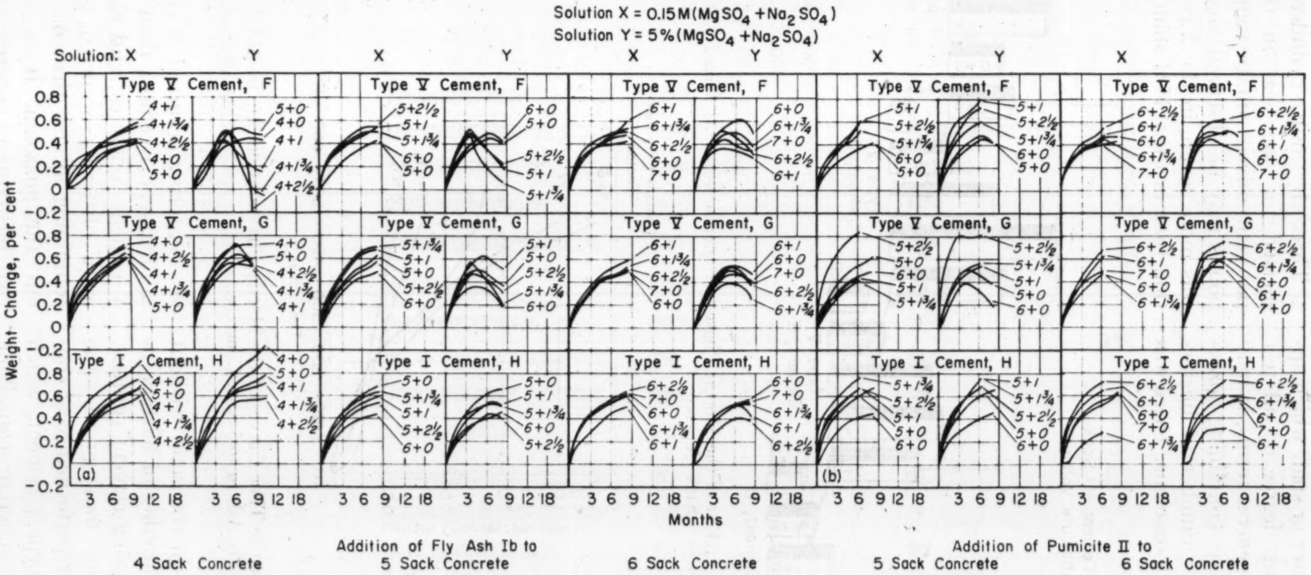

FIG. 6.—Weight Change of Concrete in 0.15*M* and 5 per cent Mixed Sulfate Solution.
(*a*) Additions of fly ash I*b* to 4, 5, and 6-sack concretes.
(*b*) Additions of pumicite II to 5 and 6-sack concrete.

teristic surface blister or spall which appeared to a varying degree on all test cylinders. In very general terms, it may be said that concretes containing pozzolans bore a greater number of blisters than those without pozzolans. There was also an indication in concretes without pozzolan that the number of blisters increased with increasing cement content. Another interesting finding was the apparently superior performance of concretes made with type I cement, which spalled less than concrete containing type V cement.

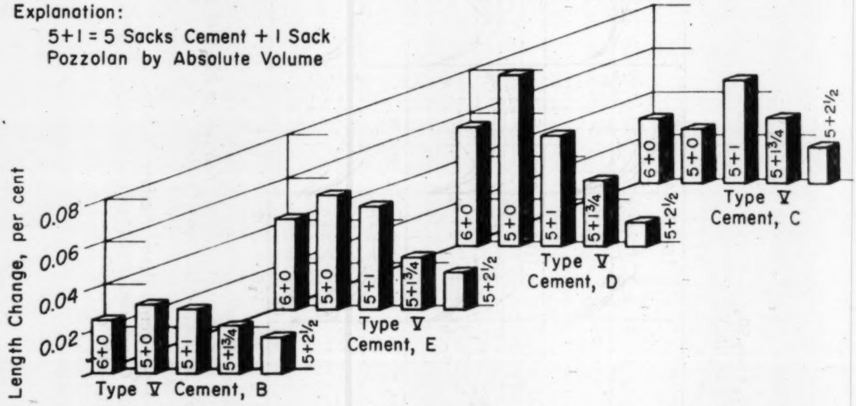

FIG. 7.—Expansion at 1 yr of the Mortar Phase of Concrete in 0.15*M* Mixed Sulfate Solution—Addition of Fly Ash I*a* to 5-Sack Concrete.

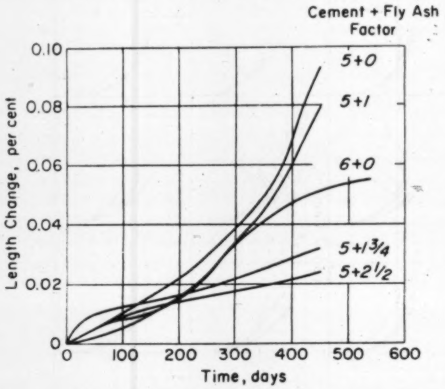

FIG. 8.—Length Change Time Curves of the Mortar Phase of Concrete in 0.15*M* Mixed Sulfate Solution—Addition of Fly Ash I*a* to 5-Sack Concrete.

Three by 6-in. cylinders were immersed for one year in 0.15*M* or 5 per cent mixed sulfate solution. Three points may be observed: first, blistering was more severe in the 5 per cent solution than in the 0.15*M* solution; second, less blistering occurred in concrete containing type I cement than on the type V cement concretes; and third, slightly more blistering occurred on concretes containing fly ash than on those without the pozzolan. It is also evident that blistering of concrete in the 0.15*M* solution was less severe than in the phase I tests. No definite reason can be seen for this although it is to be noted that different fly ash and cements were used and mix procedures were slightly modified.

A number of specimens representing a large cross-section of the tests were cut with a diamond saw and examined closely under the microscope. This revealed that the blisters or spalls were mainly a surface phenomenon and that the interior of the specimens was sound. It was also found in the petrographic examination that a film of $Mg(OH)_2$ that had formed on the concretes may have offered a resistance to infiltrating sulfate ions. The film was heaviest on concrete made with type I cements, and this may be the reason for its apparently superior performance.

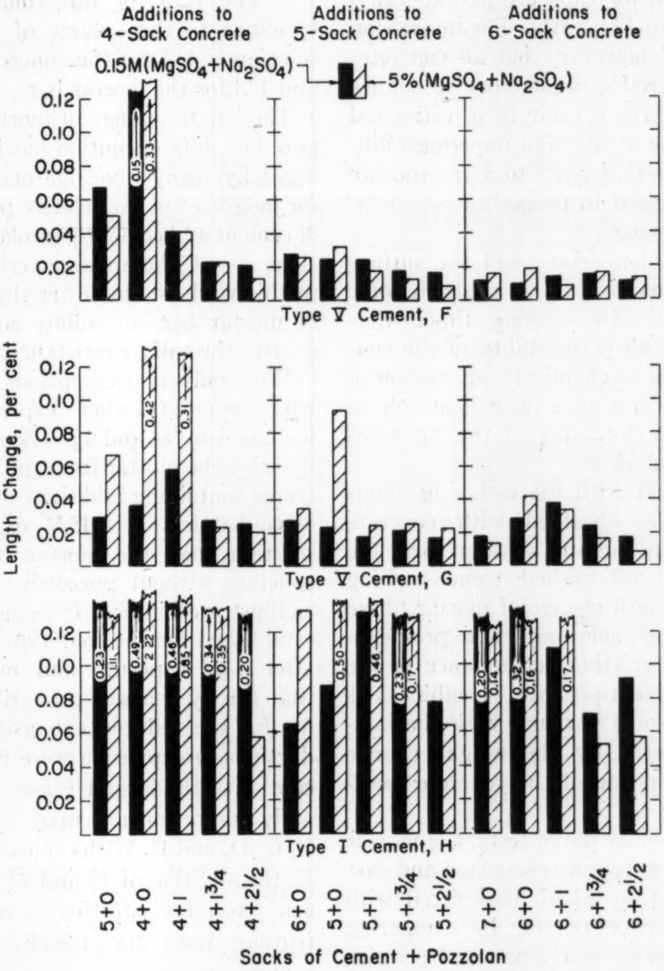

FIG. 9.—Expansion at 8 Months of the Mortar Phase of Concrete in 0.15*M* and 5 per cent Mixed Sulfate Solution—Addition of Fly Ash I*b* to 4, 5, and 6-Sack Concrete.

As with the weight change results, it was difficult to draw definite conclusions from visual ratings on the relative performance of concretes immersed in sulfate solution for exposure periods ranging from 6 to 12 months. The impression was gained, however, that all concretes to date offered a satisfactory resistance to sulfate attack, and it is estimated that because of the high impermeability of the concrete, from 3 to 5 yr exposure will be required to properly assess relative performance.

The question arises: will the surface phenomenon as it now exists become insignificant with passing time? Certainly, over-all permeability of the concrete as well as chemical composition of the paste will play a prominent role in long-term performance of the concretes in sulfate solution.

In contrast with the rather nebulous early results obtained with concrete cylinders in sulfate solution are the rather well established trends arising from the length change of mortar phase bars in sulfate solution. These provide a measure of relative performance of the various cement-pozzolan combinations that is at some variance with the indications offered by the weight change and visual rating of the concrete specimens.

In the mortar phase tests, the mortar from the concrete was screened and cast into 1 by 1 by $6\frac{1}{4}$-in. bars fitted with metal reference points. In comparing the performance of the mortar bars with the 3 by 6-in. concrete cylinders in sulfate solution the following points are important:

1. Both have the same ratio of water to cement plus pozzolan.

2. In a unit volume of concrete, cement makes up approximately $\frac{1}{10}$ the volume.

3. In a unit volume of mortar from mortar phase of concrete, cement makes up approximately $\frac{1}{5}$ the volume.

4. The ratio of the volume of the specimen to its surface is approximately $1{:}1\frac{1}{2}$ for the concrete cylinder and 1:4 for the mortar bar.

5. The ratio of the volume of the specimen to the volume of the sulfate solution is 1:1 for the concrete cylinder and 1:3 for the mortar bar.

The length change of mortar bars exposed in sulfate solution has been recognized by many investigators as a basis for judgment of the relative performance of cement and cement-pozzolan combinations (6–9).[4] The apparent criterion used in this work is: the lower the expansion of mortar bars in sulfate solution, the greater the sulfate resistance.

With only two exceptions (the series with cement C, where expansions were increased by 1- and $1\frac{3}{4}$-sack additions of fly ash), the mortar from the 5-sack concretes containing additions of fly ash I*a* expanded less in 0.15*M* mixed sulfate solution than the mortar from 5-sack concrete without pozzolan. This trend, as illustrated in Fig. 7, seems to be well established for the four type V cements after 1-yr exposure and may suggest that the fly ash was primarily responsible for the reduced expansion. The reductions in expansion were most significant with the largest ($2\frac{1}{2}$-sack) addition of fly ash for concrete made with cements B, C, D, and E. With cements B, D, and E, the addition of $1\frac{3}{4}$ and $2\frac{1}{2}$ sacks of fly ash was more effective in reducing expansion than the 1-sack addition of cement.

Except for the series containing cement C, the addition of 1 sack of pozzolan and 1 sack of cement reduced expansion by approximately the same amount. The series with cement C showed an increase in expansion with a 1-sack addition of fly ash; however, it also showed an increase in expansion for a 1-sack addition of cement, which does not conform to the general trend.

The length change - time curves for the mortar phase series with cement E are illustrated in Fig. 8. Results for 15 months exposure show in the top two curves of the chart, accelerating expansion of the mortar phase in the case of the 5-sack reference mix and concrete with 1 sack of fly ash. Mortar phase from the concrete containing 1¾ or 2½-sack additions of fly ash expanded more slowly and had not started to accelerate, as may be seen in the lower two curves of the chart. The 6-sack reference mix without fly ash held a middle course between the two extremes.

In later tests using fly ash I*b* and pumicite II in combination with three cements, some slight trends developed in the expansion of the mortar phase after short-term exposure in sulfate solution. Eight-month results for the fly ash combinations are given in Fig. 9. Results after 6½ months are shown in Fig. 10 for the pumicite combinations.

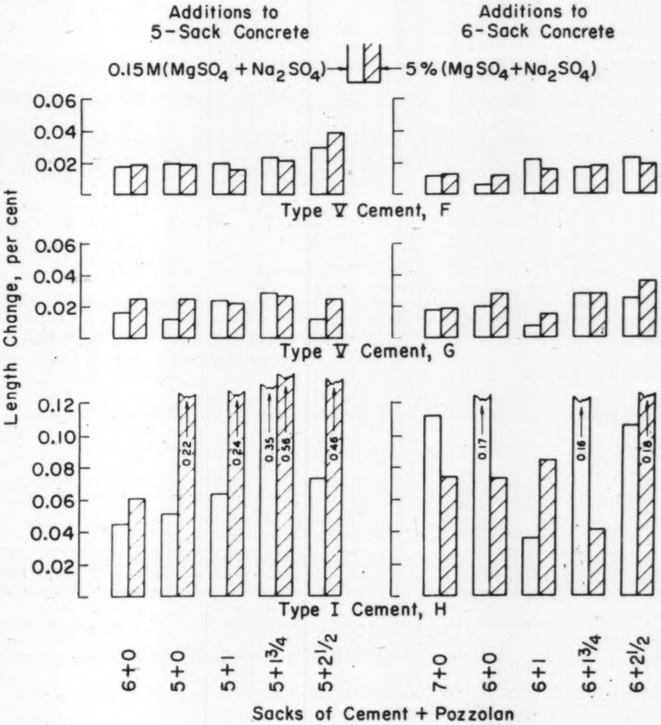

FIG. 10.—Expansion at 6½ Months of the Mortar Phase of Concrete in 0.15*M* and 5 per cent Mixed Sulfate Solution—Addition of Pumicite II to 5 and 6-Sack Concrete.

The trends that were most pronounced in the 4-sack series bear out the initial test results that show a lowered expansion of the mortar phase due to the additions of fly ash. This held true in the 0.15*M* and 5 per cent solutions with no definite trend for greater expansion in one solution or the other. The fly ash additions had a similar effect in concretes containing type I and type V cements, but the over-all performance of the two types was very different. Expansion of mortar phase bars from concretes made with type I cement was up to 20 times greater than for bars from concrete containing the type V cement.

There was also some very slight indication of the above trends in the 5- and 6-sack series with fly ash. The differences were not great at 8-month exposure with expansion for type V cements, rarely exceeding 0.03 per cent. In contrast, expansions for type I cements were in the range of 0.06 to 0.50 per cent.

In the 5- and 6-sack series with pumicite, the expansion trends, although slight, were the reverse of those obtained with the fly ash—that is, additions of the pumicite seemed to increase the expansion of the mortar phase in sulfate solution in relation to straight cement-concrete.

In some cases, results were rather erratic in the phase II tests. This was particularly noticeable in tests with type I cement, where marked differences occurred in expansions between repeat mixes of equal cement factor.

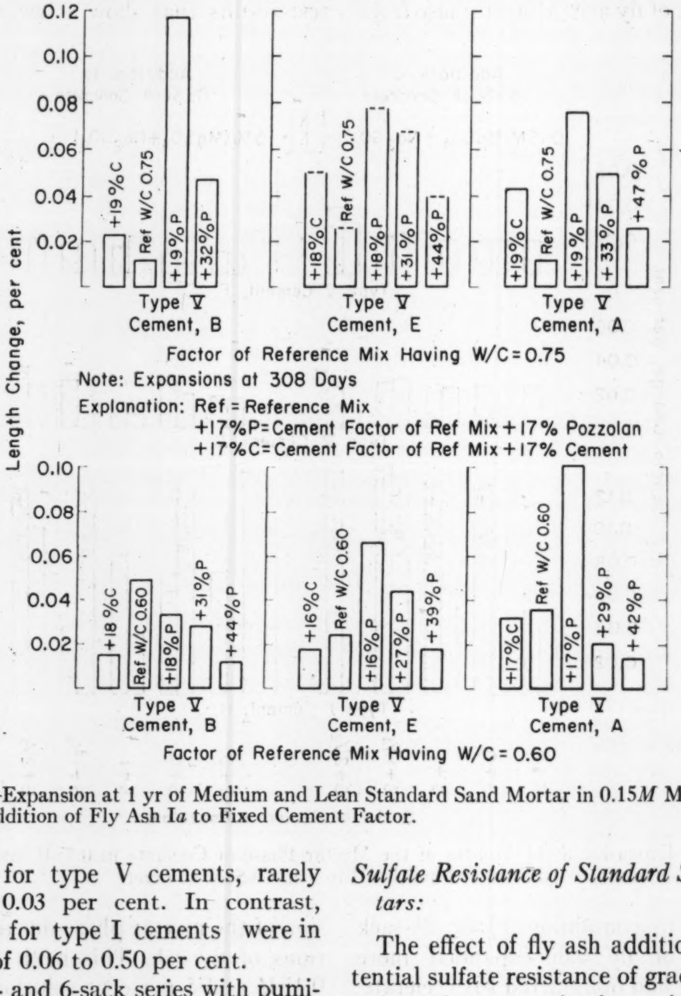

FIG. 11.—Expansion at 1 yr of Medium and Lean Standard Sand Mortar in 0.15*M* Mixed Sulfate Solution—Addition of Fly Ash I*a* to Fixed Cement Factor.

Sulfate Resistance of Standard Sand Mortars:

The effect of fly ash additions on potential sulfate resistance of graded standard sand mortars is shown in Fig. 11. Expansions at 1 yr of mortar bars in a 0.15*M* mixed sulfate solution illustrate the relative effect of additions of cement and fly ash to fixed cement factors in medium ($W/C = 0.60$) and lean ($W/C = 0.75$) mortars. Increments in the comparison mixes were on the order of 16 to 19 per cent cement and 16 to 49 per cent fly ash.

Medium mortars made with three type V cements and containing the largest additions of fly ash expanded less than the reference mortars without the pozzolan; the reductions in expansion were equal to or slightly greater than those resulting from 16 to 18 per cent added cement. With the two smaller increments of fly ash, the trend for reduced expansion was obtained in three tests out of six, and in the remaining three marked increases in expansion were recorded.

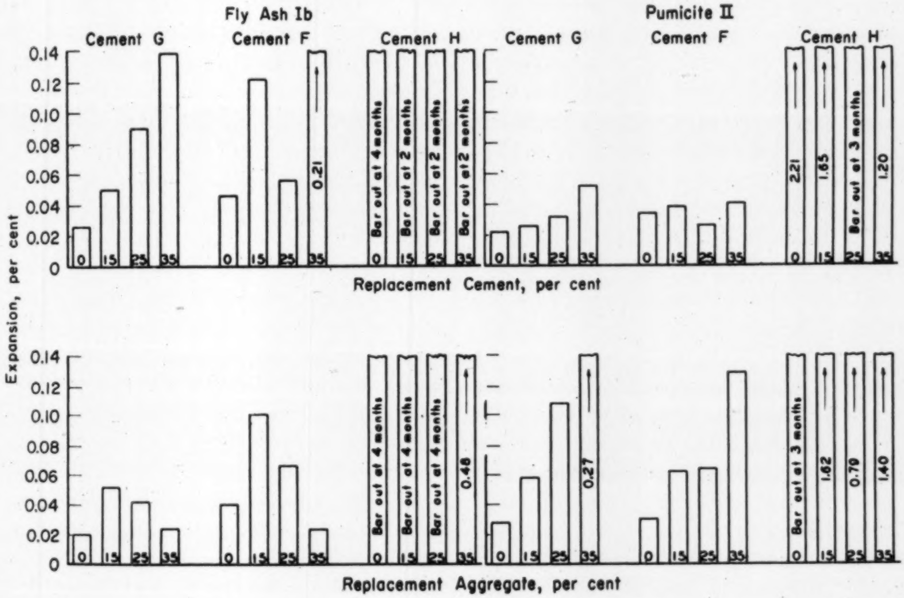

FIG. 12.—Expansion at 6 months of Lean Standard Sand Mortar in 0.15*M* Mixed Sulfate Solution—Replacement of Cement and Sand with Fly Ash I*a* and Pumicite II.

In the tests with lean mortars, the effect of fly ash was to increase expansion to a certain level with the smallest addition and reduce this level with larger increments. All expansions of mortars containing fly ash were greater than those of the reference mortars.

A surprising result was the very low 1-yr expansion of the lean reference mortars in comparison with the higher expansion of medium reference mortars. As seen in Fig. 11, the net effect of 18 and 19 per cent added cement was to increase rather than decrease expansion. Since this was contrary to the expected trend between mortars of different cement content, the possibility of an error in procedure was considered. A thorough check showed the possibility to be remote, and this was supported by compression tests of the identical mortar mixes described previously and illustrated in Fig. 4. Differences in strength level between the mortars with water-cement ratio 0.75 and 0.60 were as expected, on the basis of the water-cement ratio theory. The results might, therefore, indicate an optimum cement content in the standard sand mortars which will provide maximum sulfate resistance.

With two exceptions, replacements of fly ash I*b* for cement or sand in lean (1:6 by absolute volume) standard sand mortars resulted in increased expansion of test bars in 0.15*M* mixed solution. This is illustrated in Fig. 12, which gives values for 6 months' exposure. On the average, increases were greatest with cement replacement, this effect becoming more pronounced with increasing increments of the fly ash. The effect of sand substitution was to increase expansion to a certain level with 15 per cent replacement and reduce that level by 25 and 35 per cent replacement. With type V cement F and type I cement H, 35 per cent replacement of sand resulted in reduced expansion over that obtained in the control mixes without the fly ash.

Type I cement mortars with or without fly ash expanded rapidly in comparison with mortars containing type V cement. Most specimens cracked and failed within 4 months after immersion in sulfate solution. An exception was mortar in which 35 per cent of the sand was replaced with fly ash; specimens from this mix remained in the test at 6 months with an expansion of 0.48 per cent.

Replacements with pumicite in lean mortar produced results quite different from those obtained with fly ash. Expansion of test bars after 6 months' exposure are also shown in Fig. 12. In the type V cement mortars, substitutions of pumicite for sand produced increased expansion, the increases becoming more pronounced with larger increments of replacement. The same trend occurred but to a lesser degree with cement substitutions. In type I cement mortars, substitutions of pumicite for cement or sand reduced expansions, with the exception of 25 per cent replacement of cement. Expansion rate of all mortars containing type I cement was much more rapid than those containing type V cement.

Alkali-Aggregate Reaction:

It was not expected that the processed coarse aggregate from the plateau deposit would contribute to alkali reaction since the reactive siliceous shalestone was removed by the heavy-media process. The low percentage occurring in the processed fine aggregate gave cause for concern and resulted in detailed studies to check for any adverse action in concrete.

Three $3\frac{1}{2}$ by $4\frac{1}{2}$ by 16-in. concrete beams fitted with metal reference points were cast from each of the comparative mix series in phase II and stored in the fog room at 72 F and 100 per cent relative humidity. Two hundred specimens were made from 4-, 5-, and 6-sack concretes containing job processed aggregates, cements F, G, and H, and varying additions of fly ash I*b* and pumicite II. Alkali content of the fly ash was 3.1 per cent and ranged from 0.37 to 0.96 per cent in the cements. Length change of all specimens after storage in the fog room for 1 to $1\frac{1}{2}$ yr was low, ranging from 0.005 to 0.016 per cent. No cracking was observed and no significant differences in expansion were found between mixes with and without the pozzolan or between those containing the different cements.

Separate tests were performed to compare the volume change of specimens stored in the fog room at 72 F and 100 per cent relative humidity with specimens stored over water in sealed containers at 100 F and 100 per cent relative humidity. Concrete mixes in this series were also performed with the job processed aggregates and contained 5 sacks of cement per cu yd with varying additions of fly ash I*b*. Alkali content of the two type V cements used was

0.32 and 0.63 per cent; the fly ash contained 3.1 per cent alkalies. Three $3\frac{1}{2}$ by $4\frac{1}{2}$ by 16-in. beams were cast from each mix for fog room storage and three 3 by 3 by 11-in. beams were made for storage in the sealed containers. After an 8 to 10 month interval, no definite difference developed between expansions of specimens in the two storage conditions. Length change of all the beams was low and uniform in the range of 0.002 to 0.010 per cent. Three 1 by 1 by $11\frac{1}{4}$-in. mortar bars were also cast from the mortar phase of each mix and stored over water in the sealed containers at 100 F. Expansions of these were only slightly higher than the companion concrete specimens—in the range of 0.005 to 0.015 per cent at 8 to 10 months.

Mortar mixes were used to study the potential alkali reactivity of the processed fine aggregate. Proportions were selected on the basis of the previously described standard mortar bar test, ASTM C 227,[2] and the non-standard pozzolan addition method. For purposes of comparison, ASTM procedure was followed in the non-standard method for casting the 1 by 1 by $11\frac{1}{4}$-in. bars and storing them in sealed containers at 100 F and 100 per cent relative humidity.

Eighteen tests using monthy composite and random processed fine aggregate samples were performed in accordance with ASTM Method C 227,[2] using type I cement H with an alkali content of 0.96 per cent. The length change of 61 of the 72 bars from the tests was 0.030 to 0.043 per cent at 1 yr. These expansions were well within the limits defined in ASTM Specifications C 33 – 57[6] which state: "While the line between nonreactive and reactive combinations is not clearly defined, expansion is generally considered to be excessive if it exceeds 0.05 at 3 months or 0.10 per cent at 6 months." The remaining 11 bars cracked after 2 to 6 months storage, 7 of these fell apart and were removed from the test, 4 bars were left in the containers and attained an expansion of 0.04 to 0.07 per cent at 1 yr. An examination of the cracked bars revealed reacted siliceous shalestone particles on the failure planes and the telltale gel which accompanies alkali aggregate reaction. It was evident that considerable expansion accompanied the reaction and that in most cases the disruption was due to clusters of shalestones on the failure surface.

In the non-standard mortar bar test, varying increments of pozzolan were added to fixed cement factors in medium and lean mortars. The tests were designed to parallel the phase II concrete tests and similar materials were used, that is, job processed fine aggregate, cements F, G, and H, fly ash *Ib* and pumicite II. Length change of 240 mortar bars stored in the sealed containers was also low, ranging from 0.01 to 0.04 per cent at 1 yr. Although differences were not great, highest expansions were obtained in mortars containing cement H (alkali content 0.96 per cent).

Only the standard mortar bar test, ASTM Method C 227,[2] confirmed that siliceous shalestone in the processed fine aggregate was reactive with the alkalies in cement as revealed by cracking and formation of gel at concentrations of shalestone particles in about 15 per cent of the stored mortar bars. It is significant, however, that expansions were low and well within specified limits for the great majority of the test bars, indicating perhaps general suitability of the sand for concrete especially with cement containing less than 0.60 per cent alkalies. This is supported by the nonstandard tests of concrete and mortar with and without the additions of pozzolans where the ratio of alkalies to reactive constituent varied widely. Expansions were low and cracking did not occur during long-term storage of concrete and mortar prisms in the fog room at 72 F and 100 per cent relative humidity or over water in sealed containers at 100 F and 100 per cent relative humidity.

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[6] ASTM Specifications for Concrete Aggregates (C 33 – 59), 1959 Supplement to ASTM Book of Standards, Part 4.

Examination of old concrete structures in a damp environment and specimens in a natural exposure plot also provided evidence that a low percentage of siliceous shalestone in western aggregates did not cause serious deterioration due to alkali aggregate reaction. Slightly reacted shale particles were found in these concretes but this was unaccompanied by cracking or deterioration. As far as can be determined, cements used in the structures contained alkalies in the range of 0.40 to 0.70 per cent. In the natural exposure plot, the cements used had alkali contents in the range of 0.40 to 1.0 per cent.

Natural Exposure in Bearpaw Shale:

Relative performance of test specimens embedded in Bearpaw shale at the dam site will depend upon resistance to weathering as well as sulfate attack. Sulfate content in the soil ranges from 0.5 to 1 per cent, and specimens will be exposed to cycles of wetting and drying and freezing and thawing, with extremes of temperatures ranging between 100 F in midsummer to −50 F in winter. It is estimated that the specimens will be completely frozen for about four months in each year. The cylinders from each mix were placed in the soil with top surfaces exposed. It is expected that the first ratings will be made in July, 1962.

The system of rating is illustrated in Fig. 13.

Specimens will be rated from 1 to 6 depending upon the stage of deterioration. A rating of 1 represents perfect condition and 6 complete disintegration.

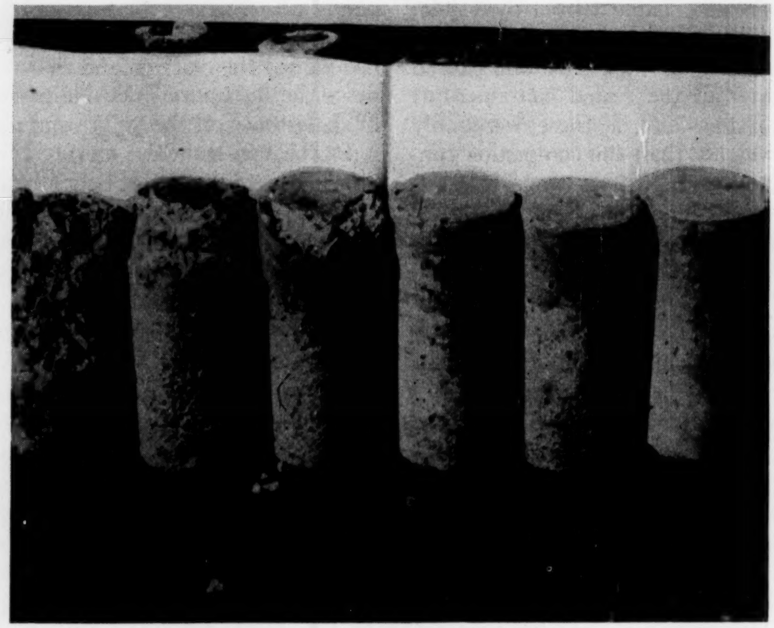

FIG. 13.—Illustration of Concrete Ratings.

While results from natural exposure will not be forthcoming for many years, it is of interest to dwell briefly on performance of other concretes to date. Tests were performed in 1952 and 1955 to evaluate the performance of concrete and concrete materials that might be used in actual construction. All mixtures contained entrained air and washed and graded aggregates from the plateau deposit. A variety of types I and V cements were used, with the cement factor in the range of $5\frac{1}{2}$ to $6\frac{1}{2}$ sacks per cu yd. After 5 and 8 yr of natural exposure, the majority of specimens had a rating of 1, with none greater than 2. Performance of concretes made with types I and V cements were about equal.

TABLE IV.—RATINGS AT 5 YR NATURAL EXPOSURE OF VOLUME-BATCHED CONCRETES.[a]

Mix Proportions, by (accurately measured) volume	Average Calculated Cement-Factor, sacks per cu yd	Ratings at 5 yr		
		Pit-Run, Gradation—Sand: Gravel		
		50:50	60:40	70:30
1:4.........	6¼	1	1	2
1:5.........	5¼	2	2	3
1:6.........	4½	3	3	[b]
1:7.........	3¾	5	6	6
1:8.........	3¼	6	6	6

[a] Type I cement—clean regraded pit-run granular material.

[b] Specimens lost.

In the winter of 1955, a series of tests was performed during a concrete inspectors course to study volume batching methods as related to very small isolated projects. Since the tests were performed under closely controlled laboratory conditions, specimens were cast for natural exposure. Three different pit-run gradations were used, and the mix proportions ranged from 1:4 to 1:8. Water was added to maintain slump between 3 and 4 in., and mixes were repeated until this range was obtained. The average ratings of specimens determined after 5-yr exposure are given in Table IV. The concretes were not representative of the carefully designed and graded mixtures that will be used in concrete construction, yet their relative performance illustrates the importance of adequate cement for durability and the harmful effect of high porosity—in this case achieved by over-sanding.

The environment of tunnel concrete will be much different from the conditions in the exposure plot. There will be little chance for evaporation and concentration of salts, since the tunnels will be surrounded by soil and filled with water. It is expected, therefore, that the degree of attack will be small in comparison with structures such as slab resting in the soil and exposed to the elements.

The pressure section of a test drift constructed in Bearpaw shale at the site in 1950 provided conditions quite similar to those expected for tunnel concrete. The drift was uncovered during excavation work at the dam in 1961 and the concrete slabs found to be in excellent condition. The concrete was made with a high C_3A type III cement and contained a minimum of 7 sacks per cu yd. Careful control used in design proportioning and placement of the concrete resulted in a highly impermeable material which may be the prime reason for the satisfactory performance.

SUMMARY AND CONCLUSIONS

The addition of fly ash to fixed cement factors in concrete resulted in lowered water requirement, improved workability, and satisfactory early strength gain. Improved workability was also obtained with a pumicite; however, water requirement was increased markedly and compressive strengths at ages up to 6 months were reduced in relation to the reference concrete without the pozzolan.

No conclusions on relative performance of specimens in sulfate solution are attempted at this time since it is possible that the arbitrary procedures may give misleading results, especially in the early period. Even in the long-term analysis, it will be difficult to relate the results of the accelerated tests to the environment of structures at the South Saskatchewan River Dam. Ground waters at the site contain sulfate in quantities which warrant the use of type V cement, and yet the water sample with highest sulfate content contains only one half as much SO_4 as the $0.15M$ mixed solution of $MgSO_4$ and Na_2SO_4 used in the laboratory. Magnesium content is a predominant factor in deterioration of type V cement concrete, and the $0.15M$ solution contains about 7 times as much magnesium as the ground water sample highest in magnesium. Since the tunnels will be surrounded by soil and filled with water, there will be little chance for evaporation and concentration of salts. The exposure conditions in the laboratory are very much more severe than for the tunnels, due to the unnatural conditions of total immersion and high ratio of exposed surface to the total mass of the small specimens.

Considering these factors and on the basis of fairly satisfactory performance to date of laboratory specimens in $0.15M$ mixed sulfate solution, a tentative forecast has been made. Concrete that is well designed, manufactured and placed and containing type V cement, processed aggregates from the site, cement factor in the range of 5 to 6 sacks per cu yd with or without additions of fly ash should give satisfactory long-term performance in the tunnels.

The heavy-media process was specified to remove lightweight deleterious material from the coarse aggregate at the dam site. One of the lightweight constituents—a siliceous shalestone—contained opal and was found to be reactive with the alkalies in cement. A low percentage of the shalestone in the processed fine aggregate resulted in some cracking of mortar bars stored at 100 F and 100 per cent relative humidity in accordance with ASTM C 227.[2] Expansion of the majority of test bars was low, indicating suitability of the sand for concrete construction.

Opal content in a typical concrete mix was estimated to be about 0.3 per cent, and therefore alkali reactivity studies were performed to check for an adverse action in concrete. Concrete and mortar beams from comparative mix series were placed in 100 per cent relative humidity at 72 F and 100 F. The tests embraced several cements containing 0.4 to 1 per cent alkalies, fly ash containing 3 per cent alkalies, pumicite containing 0.8 per cent alkalies and combinations of the materials in rich, medium, and lean concretes and mortars. The ratio of alkalies to reactive constituent varied widely and yet no abnormal expansion of the test bars occurred in periods ranging to 18 months. While it is perhaps too early to reach definite conclusions, the results are at least reassuring with respect to the suitability of the processed aggregates.

Field observations of existing concrete structures in western Canada and naturally exposed concrete specimens have revealed the presence of slightly reacted shalestone particles but no deleterious effects arising from alkali aggregate reaction. The observations have also shown that the many failures attributed to sulfate attack can be traced to a very permeable concrete resulting from faulty construction practice. It is not certain that the failures can be attributed entirely to the type I cement, which was invariably used. It is doubtful if even the available type V cements would have prevented deterioration in these lean permeable concretes.

Permeability is considered the key to

high sulfate resistance in hydraulic structures appurtenant to the South Saskatchewan River Dam. Careful control in aggregate processing and concreting techniques should provide a highly impermeable concrete. The improved mix characteristics and possibly long-term impermeability produced by a pozzolan such as the fly ash tested would be an asset in production of a dense concrete.

A low C_3A type V cement will be used to achieve maximum chemical stability and the compatibility of pozzolans with such a cement is still a matter under study.

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(3) "The Condition of Field Specimens of Concrete Exposed to Alkali Soils and Waters Examined in December, 1927," from a report of the Committee on the Deterioration of Concrete in Alkali Soils, Engineering Inst. of Canada, *The Engineering Journal*, Vol. XI, No. 3, March 1928, p. 184.

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(5) E. Stanton, "Durability of Concrete Exposed to Sea Water and Alkali Soils—California Experience," *Proceedings*, Am. Concrete Inst., Vol. 44, p. 821 (1948).

(6) T. Thorvaldson and V. A. Vigfussen, "The Effect of Steam Treatment of Portland Cement Mortars on Their Resistance to Sulfate Action," *The Engineering Journal*, Engineering Inst. of Canada, Vol. XI, No. 3, March 1928, p. 174.

(7) T. Thorvaldson, "Chemical Aspects of the Durability of Cement Products," Proceedings of the Third International Symposium of the Chemistry of Cement, London, p. 436 (1952).

(8) D. Miller and P. Manson, "Long-Time Tests of Concretes and Mortars Exposed to Sulfate Waters," *Technical Bulletin 194*, University of Minnesota, May 1951.

(9) D. Miller and C. G. Snyder, "Appendix III Report on Comparative Short-Time Tests for Sulfate Resistance of 121 Commercial Cements," *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 165 (1945).

CORRELATION OF FLEXURAL AND COMPRESSIVE STRENGTHS OF CONCRETES AND MORTARS*

By Ivan L. Lynn[1] and Kenneth E. Palmer[2]

Synopsis

Statistical correlations of various flexural and compressive strengths of laboratory-prepared concretes and mortars using 37 type I, II, and III portland cements from different parts of the United States are reported.

The investigation covered mortar compressive strengths for regular 2 by 2-in. cubes and 40 by 40-mm modified cubes, and flexural strengths of 40 by 40 by 160-mm prisms. Concrete compressive strengths were for 3 by 6-in. cylinders, and the flexural strengths were for midpoint loading of 3 by 3-in, prisms with a 9-in. span. The concrete consisted of both 4 and 6 sacks of cement per cubic yard. Test ages were 3, 7, 28, and 90 days, and type III cements included 1-day tests.

Highly significant correlation equations for several strength relationships are presented for each of the cement types studied and for a combination of the three cement types.

Often it is desirable to predict the strength of either concretes or mortars when the results from one or the other of these materials have been established by actual testing. Since strength results for both mortars and concretes increase with age, it appears logical that there should exist a correlation between the strength results for these two materials

Kesler (**1,2**)[3] has shown that relationships of concrete strengths, as determined by the compressive tests of cylinders or modified cubes and the modulus of rupture for concrete beams, will produce good correlation. Others have presented data for both mortars and concretes, primarily in tabulated form, such as the cooperative test work of the Working Committee on Strength of ASTM Committee C-1 on Cement (**3**) and a report by Swayze (**4**).

The ASTM has outlined compressive strength test procedures for mortars by the use of 2 by 2-in. cubes or 40 by 40-mm modified cubes. Mortar flexural tests are made on the 40 by 40 by 160-mm bars. Standard methods are also available for determining concrete compressive and flexural strength. This paper presents statistical correlations of these various strength tests—mortar versus mortar, concrete versus mortar, and concrete versus concrete—by means of regression equations.

Regression equations for twelve different relationships and their correlation coefficients are presented. An analysis of the precision of each of the equa-

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

TABLE I.—TYPE I CEMENTS: MORTAR AND CONCRETE TEST RESULTS.

Cement	Mortar Water: Cement Ratio	Concrete Water: Cement Ratio		Test Age, days	Mortar Specimen Strength, psi			Concrete Specimen Strength, psi			
								6-Sack Mixes		4-Sack Mixes	
		6-Sack	4-Sack		2 by 2-in. Cube Compression	40 by 40 by 160-mm Flexure	40 by 40-mm Modified Cube Compression	3 by 6-in. Cylinder Compression	3 by 3-in. Flexure	3 by 6-in. Cylinder Compression	3 by 3-in. Flexure
A	0.476	0.495	0.769	3	2840	580	2950	3020	690	1130	350
				7	4030	790	4120	4350	870	1820	520
				28	5490	930	5830	5890	900	2960	720
				90	6470	1040	6220	7010	1020	3290	820
B	0.490	0.511	0.769	3	1790	520	2540	2550	620	1020	350
				7	2710	720	3700	3990	810	1750	600
				28	4170	900	5020	5450	900	2820	730
				90	4600	970	5560	6040	1190	3220	770
C	0.486	0.505	0.769	3	2490	420	2040	2390	620	980	290
				7	3610	540	2950	3600	760	1670	490
				28	4970	730	4060	4920	1030	2620	750
				90	5480	950	4750	5680	1150	2940	810
D	0.500	0.511	0.777	3	2030	470	2220	2300	560	930	310
				7	3050	640	3220	3400	770	1540	500
				28	4230	870	4350	4980	930	2640	700
				90	4910	1020	4830	5590	1060	3140	760
E	0.505	0.505	0.785	3	2130	440	2990	2320	570	920	300
				7	3270	660	3490	3750	770	1510	520
				28	5030	1020	5110	5250	1020	2590	710
				90	6100	980	4610	6120	1220	3260	880
F	0.473	0.495	0.769	3	2630	550	2740	2930	710	960	340
				7	4370	750	4430	4820	910	2000	520
				28	6340	1100	6280	6260	1020	3290	770
				90	7800	1060	6340	7010	1090	3310	750
G	0.510	0.511	0.769	3	1680	400	2100	1980	490	770	270
				7	3470	650	3430	3880	790	1550	470
				28	4830	910	4830	5840	900	2810	750
				90	5380	980	5270	6090	1060	3420	870
H	0.500	0.511	0.774	3	2400	570	2680	2860	690	1210	440
				7	3660	650	3890	4420	840	1960	520
				28	5560	870	5870	6070	1010	3220	730
				90	6170	1020	6340	6970	1190	3660	880
J	0.480	0.505	0.769	3	3370	640	2790	3400	740	1340	510
				7	4810	790	4730	4810	890	2100	590
				28	5980	940	5250	6350	1040	3530	740
				90	6230	950	5730	6710	1110	3690	790
K	0.500	0.522	0.785	3	2440	540	2460	2690	660	1210	390
				7	3610	810	4040	4060	770	1810	520
				28	5180	930	4870	5230	980	2720	690
				90	5780	1060	5890	5760	1170	3030	760
L	0.486	0.500	0.769	3	2700	540	2900	2450	610	940	250
				7	4670	770	4830	4590	900	1910	530
				28	7190	1010	7080	6780	1000	3520	780
				90	7870	1040	6950	7120	1270	3680	820

TABLE I.—*Continued.*

Cement	Mortar Water: Cement Ratio	Concrete Water: Cement Ratio		Test Age, days	Mortar Specimen Strength, psi			Concrete Specimen Strength, psi			
								6-Sack Mixes		4-Sack Mixes	
		6-Sack	4-Sack		2 by 2 in-Cube Compression	40 by 40 by 160-mm Flexure	40 by 40-mm Modified Cube Compression	3 by 6-in. Cylinder Compression	3 by 3-in. Flexure	3 by 6-in. Cylinder Compression	3 by 3-in. Flexure
M	0.493	0.495	0.769	3	2530	530	2640	2980	700	1200	260
				7	3950	760	4130	4510	820	1840	560
				28	5760	850	5830	6010	1060	3140	720
				90	6300	1030	6450	6990	1190	3610	810
N	0.493	0.511	0.769	3	2800	570	2920	2670	620	1190	380
				7	4290	810	4230	4290	840	2060	560
				28	6230	1030	6190	6080	1050	3270	760
				90	6870	1060	7330	6940	1180	3740	840
O	0.493	0.495	0.769	3	2510	520	2750	2610	620	1070	250
				7	4290	770	4320	4400	820	1930	480
				28	6100	970	5900	6470	1030	3440	760
				90	6310	1030	6680	7190	1100	3960	900
P	0.470	0.511	0.768	3	2610	550	2330	2260	550	1100	350
				7	3930	780	3980	3920	820	1790	570
				28	6540	980	6040	5770	970	3280	800
				90	7230	1150	7310	6840	1240	3770	890

tions is made by the standard error of estimate for the dependent variable in each of the equations.

MATERIALS AND TEST PROCEDURES

The cements for this investigation consisted of 15 type I, 13 type II, and 9 type III portland cements from various parts of the United States. These were received in connection with several projects undertaken by the Research Department of the Ideal Cement Co., and the data compiled in this report were obtained from these individual laboratory projects conducted over a period of approximately one year. All concrete and mortar tests were performed by the same group of laboratory personnel and with the same sets of laboratory facilities. Strength tests were made at the ages of 3, 7, 28, and 90 days for all cements, and included 1-day tests for the type III portland cements.

The investigation determined the mortar compressive strengths of 2 by 2-in. cubes (ASTM Method of Test C 109[4]) and 40 by 40-mm modified cubes (ASTM Method of Test C 349[5]). Mortar flexural strengths were determined by using 40 by 40 by 160-mm prisms (ASTM Method of Test C 348[6]). Concrete compressive strengths were determined on 3 by 6-in. cylinders (ASTM Method of Test C 39[7]), and

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[4] Method of Test for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. Cube Specimens) (C 109 – 58), 1958 Book of ASTM Standards, Part 4, p. 124.

[5] Method of Test for Compressive Strength of Hydraulic Cement Mortars (Using Portions of Prisms Broken in Flexure) (C 349 – 59 T) 1959 Supplement to Book of ASTM Standards, Part 4, p. 7.

[6] Method of Test for Flexural Strength of Hydraulic Cement Mortars (C 348 – 57 T), 1958 Book of ASTM Standards, Part 4, p. 156.

[7] Method of Test for Compressive Strength of Molded Concrete Cylinders (C 39 – 59), 1958 Book of ASTM Standards, Part 4, p. 655.

TABLE II.—TYPE II CEMENTS: MORTAR AND CONCRETE TEST RESULTS.

Cement	Mortar Water: Cement Ratio	Concrete Water: Cement Ratio		Test Age, days	Mortar Specimen Strength, psi			Concrete Specimen Strength, psi			
								6-Sack Mixes		4-Sack Mixes	
		6-Sack	4-Sack		2 by 2-in. Cube Compression	40 by 40 by 160-mm Flexure	40 by 40-mm Modified Cube Compression	3 by 6-in. Cylinder Compression	3 by 3-in. Flexure	3 by 6-in. Cylinder Compression	3 by 3-in. Flexure
A	0.477	0.495	0.769	3	1850	340	1660	2040	500	730	290
				7	2540	510	2730	2800	610	1240	330
				28	3830	760	3870	4200	870	1810	610
				90	5510	1020	5690	5450	1190	2430	690
B	0.490	0.511	0.769	3	2110	470	2180	2380	580	960	300
				7	3340	590	3220	3770	800	1610	470
				28	5690	910	5610	5900	970	2900	780
				90	7040	1100	6920	6730	1190	3430	820
C	0.482	0.511	0.769	3	1950	400	1980	1860	500	760	240
				7	2780	540	2670	2740	680	1170	430
				28	4660	830	4380	4630	970	2140	610
				90	5570	960	5330	5300	1150	2570	730
D	0.473	0.500	0.760	3	1990	400	2090	1860	440	660	200
				7	3170	620	3370	3270	730	1330	380
				28	5990	900	5930	5870	950	2780	610
				90	6900	1060	6010	6440	1040	3350	760
E	0.505	0.489	0.769	3	1970	410	2010	2260	590	830	270
				7	3810	590	3230	3590	800	1430	380
				28	5250	860	4950	5870	1030	2720	690
				90	5960	960	5600	7040	1230	3230	800
F	0.476	0.495	0.769	3	2380	490	2210	2270	570	710	270
				7	3200	590	2880	3500	740	1390	380
				28	5300	840	4850	5210	1040	2620	720
				90	6180	1080	6170	6190	1220	3110	700
G	0.485	0.505	0.769	3	2140	460	2380	2200	550	810	270
				7	3500	680	3690	3890	860	1520	460
				28	5360	920	5810	6030	890	2780	670
				90	6550	1030	5610	6350	1040	3180	810
H	0.480	0.516	0.774	3	1910	420	1920	1880	480	710	220
				7	2540	540	2910	2880	690	1140	440
				28	4410	860	4750	4970	940	2200	680
				90	6180	1090	6060	5680	1070	2940	710
J	0.470	0.495	0.769	3	2560	510	2500	2490	570	790	260
				7	3800	770	3710	3950	830	1320	410
				28	6100	1020	5640	6420	1060	2880	700
				90	7360	1110	6890	7190	1160	3470	840
K	0.500	0.511	0.769	3	2130	560	2430	2170	550	880	260
				7	2910	630	3310	3410	780	1430	390
				28	5180	870	5390	5270	1010	2490	700
				90	6480	1020	6100	6360	1200	3280	930
M	0.473	0.495	0.769	3	2240	500	2350	2570	600	780	270
				7	3520	650	3300	3840	780	1450	410
				28	5770	960	5370	6010	1010	2520	650
				90	6230	1080	6260	6780	1140	3260	790

TABLE II.—*Continued.*

Cement	Mortar Water: Cement Ratio	Concrete Water: Cement Ratio		Test Age, days	Mortar Specimen Strength, psi			Concrete Specimen Strength, psi			
								6-Sack Mixes		4-Sack Mixes	
		6-Sack	4-Sack		2 by 2-in. Cube Compression	40 by 40 by 160-mm Flexure	40 by 40-mm Modified Cube Compression	3 by 6-in. Cylinder Compression	3 by 3-in. Flexure	3 by 6-in. Cylinder Compression	3 by 3-in. Flexure
O..........	0.493	0.495	0.769	3	1910	440	2160	1940	490	770	230
				7	3510	620	3460	3650	810	1570	460
				28	5640	910	5840	5980	930	3020	700
				90	6490	1030	6600	7160	1310	3630	890
P..........	0.460	0.500	0.769	3	2690	540	2950	2530	640	1010	340
				7	3780	750	3900	3720	880	1440	440
				28	5780	1050	5870	5650	1060	2650	760
				90	6770	1110	6690	6450	1190	3320	840

concrete flexural tests were made on 3 by 3 by 17-in. prisms by center point loading using a 9-in. span (ASTM Method of Test C 293[8]). The making and curing of the concrete test specimens were performed in accordance with ASTM Method C 192,[9] with the exception of using the size of specimens denoted above and an internal vibrator for compaction.

The concretes were non-air-entrained and contained $\frac{3}{4}$-in. maximum size aggregate from Elgin, Ill. The sand and gravel gradations were constant for each mix. The concrete slumps were maintained at 3 ± $\frac{1}{2}$ in.

Tabulations of the test results and water-cement ratios for the mortars and concretes are given in Tables I, II, and III for the types I, II, and III cements, respectively. The strength results shown are the averages obtained from three specimens, except the concrete flexural results, which are the averages of two tests.

STATISTICAL CORRELATION

From the laboratory-developed strength data, single straight-line regression equations of the type $Y = a + bX$ were computed by means of the "least squares method" (5) for each of the cement types and as a unit by using the three cement types combined. Regression equations and correlation coefficients for the following 12 relationships are tabulated in Table IV:

1. Mortar cube compressive strength (2-in.) versus mortar flexural strength (40-mm),
2. Modified cube compressive strength (40-mm) versus mortar cube compressive strength (2-in.),
3. Modified cube compressive strength (40-mm) versus mortar flexural strength (40-mm),
4. Concrete compressive strength (6-sack) versus mortar cube compressive strength (2-in.),
5. Concrete flexural strength (6-sack) versus mortar cube compressive strength (2-in.),
6. Concrete flexural strength (6-sack) versus mortar flexural strength (40-mm),
7. Concrete compressive strength (6-sack) versus modified cube compressive strength (40-mm),
8. Concrete compressive strength (6-sack) versus mortar flexural strength (40-mm),
9. Concrete compressive strength (6-sack) versus concrete flexural strength (6-sack),
10. Concrete compressive strength (4-sack) versus concrete flexural strength (4-sack),

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[8] Method of Test for Flexural Strength of Concrete (Using Simple Beam with Center-Point Loading) (C 293 – 59), 1958 Book of ASTM Standards, Part 4, p. 670.

[9] Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Laboratory (C 192 – 59), 1959 Supplement to Book of ASTM Standards, Part 4, p. 96.

TABLE III.—TYPE III CEMENTS: MORTAR AND CONCRETE TEST RESULTS.

Cement	Mortar Water: Cement Ratio	Concrete Water: Cement Ratio		Test Age, days	Mortar Specimen Strength, psi			Concrete Specimen Strength, psi			
								6-Sack Mixes		4-Sack Mixes	
		6-Sack	4-Sack		2 by 2-in. Cube Compression	40 by 40 by 160-mm Flexure	40 by 40-mm Modified Cube Compression	3 by 6-in. Cylinder Compression	3 by 3-in. Flexure	3 by 6-in. Cylinder Compression	3 by 3-in. Flexure
A	0.507	0.511	0.769	1	2180	480	2170	1980	440	710	220
				3	3460	690	3420	3660	680	1590	430
				7	4800	780	4610	5060	890	2440	570
				28	6120	1020	5710	6480	970	3770	840
				90	6430	1070	6510	7170	1180	4220	900
B	0.490	0.511	0.769	1	1770	460	1990	1720	440	660	180
				3	4130	760	4350	4290	770	1920	510
				7	5480	890	5370	5370	890	2660	630
				28	7410	1140	6910	6840	1030	3890	820
				90	7440	1080	7340	7300	1160	4340	980
F	0.500	0.511	0.769	1	2430	510	2420	1660	590	830	330
				3	4370	760	4250	4350	760	1940	500
				7	5540	900	5530	5670	830	2780	650
				28	6890	1170	6730	6560	1010	3650	800
				90	7030	1030	6630	6860	1160	3750	860
G	0.486	0.505	0.769	1	1570	370	1610	1310	360	450	160
				3	3070	580	3960	3040	720	1310	420
				7	4760	720	4530	5300	860	2390	580
				28	6300	990	6120	6190	1070	3640	740
				90	6530	950	6320	7040	1140	3950	850
J	0.500	0.516	0.802	1	2470	560	2430	2020	520	580	220
				3	4300	820	3990	3720	730	1500	410
				7	5700	1020	5540	5110	810	2380	590
				28	7330	1170	6410	7000	940	3770	760
				90	7260	1090	6850	7370	1170	3870	870
K	0.495	0.511	0.760	1	1670	410	1690	1560	350	540	130
				3	3990	700	3600	3960	820	1720	490
				7	5480	850	5510	5560	910	2810	660
				28	7380	990	6630	6880	1120	4140	810
				90	7200	980	6860	6980	1130	4330	900
L	0.500	0.527	0.769	1	3030	600	3100	2070	430	780	260
				3	5330	800	4950	4530	830	2010	500
				7	6760	890	6400	6280	900	3410	700
				28	8630	1050	7470	7030	1010	4800	840
				90	9040	1030	7610	7330	1160	5030	930
N	0.507	0.511	0.769	1	2030	420	1840	1510	430	550	170
				3	4400	790	4490	4080	730	1720	480
				7	6160	900	5560	5710	860	2670	640
				28	7740	1050	7260	7120	1010	4080	860
				90	7560	1180	7420	7670	1290	4280	920
O	0.466	0.495	0.769	1	1910	430	1920	1600	440	520	190
				3	3840	700	3630	3660	730	1330	380
				7	5490	870	5450	5450	850	2290	580
				28	7410	990	6670	6660	990	4060	830
				90	7690	1110	7320	7710	1100	4430	870

TABLE IV.—REGRESSION EQUATIONS AND CORRELATION COEFFICIENTS

Equation Number[a]	Equation	Correlation Coefficient, r
1-C	2-in. mortar cube compressive strength = −1474 + 7.692 (40-mm mortar flexural strength)	0.947
1-I	2-in. mortar cube compressive strength = −1395 + 7.390 (40-mm mortar flexural strength)	0.928
1-II	2-in. mortar cube compressive strength = −1100 + 7.107 (40-mm mortar flexural strength)	0.979
1-III	2-in. mortar cube compressive strength = −1817 + 8.458 (40-mm mortar flexural strength)	0.952
2-C	40-mm modified cube compressive strength = 457 + 0.8825 (2-in. mortar cube compressive strength)	0.977
2-I	40-mm modified cube compressive strength = 594 + 0.8659 (2-in. mortar cube compressive strength)	0.956
2-II	40-mm modified cube compressive strength = 308 + 0.9154 (2-in. mortar cube compressive strength)	0.985
2-III	40-mm modified cube compressive strength = 386 + 0.8817 (2-in. mortar cube compressive strength)	0.989
3-C	40-mm modified cube compressive strength = −1048 + 7.044 (40-mm mortar flexural strength)	0.961
3-I	40-mm modified cube compressive strength = −940 + 6.806 (40-mm mortar flexural strength)	0.943
3-II	40-mm modified cube compressive strength = −796 + 6.634 (40-mm mortar flexural strength)	0.984
3-III	40-mm modified cube compressive strength = −1352 + 7.620 (40-mm mortar flexural strength)	0.961
4-C	6-sack concrete compressive strength = 374 + 0.9354 (2-in. mortar cube compressive strength)	0.967
4-I	6-sack concrete compressive strength = 604 + 0.9256 (2-in. mortar cube compressive strength)	0.961
4-II	6-sack concrete compressive strength = 238 + 0.9720 (2-in. mortar cube compressive strength)	0.983
4-III	6-sack concrete compressive strength = −38 + 0.9621 (2-in. mortar cube compressive strength)	0.973
5-C	6-sack concrete flexural strength = 354 + 0.1113 (2-in. mortar cube compressive strength)	0.893
5-I	6-sack concrete flexural strength = 389 + 0.1124 (2-in. mortar cube compressive strength)	0.914
5-II	6-sack concrete flexural strength = 304 + 0.1309 (2-in. mortar cube compressive strength)	0.948
5-III	6-sack concrete flexural strength = 254 + 0.1127 (2-in. mortar cube compressive strength)	0.931
6-C	6-sack concrete flexural strength = 130 + 0.9317 (40-mm mortar flexural strength)	0.920
6-I	6-sack concrete flexural strength = 170 + 0.9081 (40-mm mortar flexural strength)	0.927
6-II	6-sack concrete flexural strength = 138 + 0.9582 (40-mm mortar flexural strength)	0.956
6-III	6-sack concrete flexural strength = 16 + 0.9936 (40-mm mortar flexural strength)	0.923
7-C	6-sack concrete compressive strength = −28 + 1.042 (40-mm modified cube compressive strength)	0.982
7-I	6-sack concrete compressive strength = 170 + 1.024 (40-mm modified cube compressive strength)	0.963
7-II	6-sack concrete compressive strength = −5 + 1.042 (40-mm modified cube compressive strength)	0.979
7-III	6-sack concrete compressive strength = −442 + 1.088 (40-mm modified cube compressive strength)	0.981

TABLE IV.—*Continued.*

Equation Number[a]	Equation	Correlation Coefficient, r
8-C	6-sack concrete compressive strength = −1270 + 7.528 (40-mm mortar flexural strength)	0.958
8-I	6-sack concrete compressive strength = −1073 + 7.321 (40-mm mortar flexural strength)	0.954
8-II	6-sack concrete compressive strength = −857 + 6.943 (40-mm mortar flexural strength)	0.967
8-III	6-sack concrete compressive strength = −2042 + 8.442 (40-mm mortar flexural strength)	0.961
9-C	6-sack concrete compressive strength = −1629 + 7.290 (6-sack concrete flexural strength)	0.939
9-I	6-sack concrete compressive strength = −1850 + 7.398 (6-sack concrete flexural strength)	0.944
9-II	6-sack concrete compressive strength = −1508 + 6.838 (6-sack concrete flexural strength)	0.955
9-III	6-sack concrete compressive strength = −1653 + 7.883 (6-sack concrete flexural strength)	0.966
10-C	4-sack concrete compressive strength = −568 + 4.960 (4-sack concrete flexural strength)	0.964
10-I	4-sack concrete compressive strength = −602 + 4.853 (4-sack concrete flexural strength)	0.965
10-II	4-sack concrete compressive strength = −368 + 4.370 (4-sack concrete flexural strength)	0.976
10-III	4-sack concrete compressive strength = −629 + 5.445 (4-sack concrete flexural strength)	0.983
11-C	6-sack concrete compressive strength = 1188 + 1.528 (4-sack concrete compressive strength)	0.976
11-I	6-sack concrete compressive strength = 1090 + 1.580 (4-sack concrete compressive strength)	0.984
11-II	6-sack concrete compressive strength = 904 + 1.761 (4-sack concrete compressive strength)	0.988
11-III	6-sack concrete compressive strength = 1283 + 1.427 (4-sack concrete compressive strength)	0.976
12-C	6-sack concrete flexural strength = 299 + 0.9857 (4-sack concrete flexural strength)	0.949
12-I	6-sack concrete flexural strength = 310 + 0.9690 (4-sack concrete flexural strength)	0.940
12-II	6-sack concrete flexural strength = 290 + 1.0660 (4-sack concrete flexural strength)	0.957
12-III	6-sack concrete flexural strength = 269 + 0.9685 (4-sack concrete flexural strength)	0.975

[a] Code for equation numbers:
C = Type I, II, and III cements combined ($n = 157$),
I = Type I cements ($n = 60$),
II = Type II cements ($n = 52$), and
III = Type III cements ($n = 45$).

11. Concrete compressive strength (6-sack) versus concrete compressive strength (4-sack), and
12. Concrete flexural strength (6-sack) versus concrete flexural strength (4-sack).

The correlation coefficient r is a statistical term used to measure the spread of the test results about a regression line. The values of r can range from +1 to −1, with the plus and minus signs indicating the direction of the slope of the regression line. If the correlation

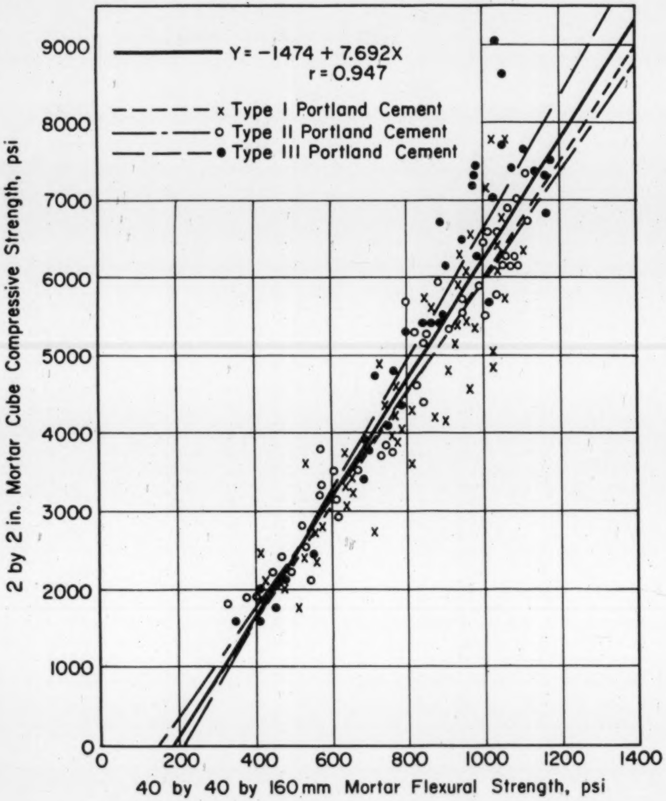

Fig. 1.—Line of Regression of Mortar Cube Compressive Strength on Mortar Flexural Strength.

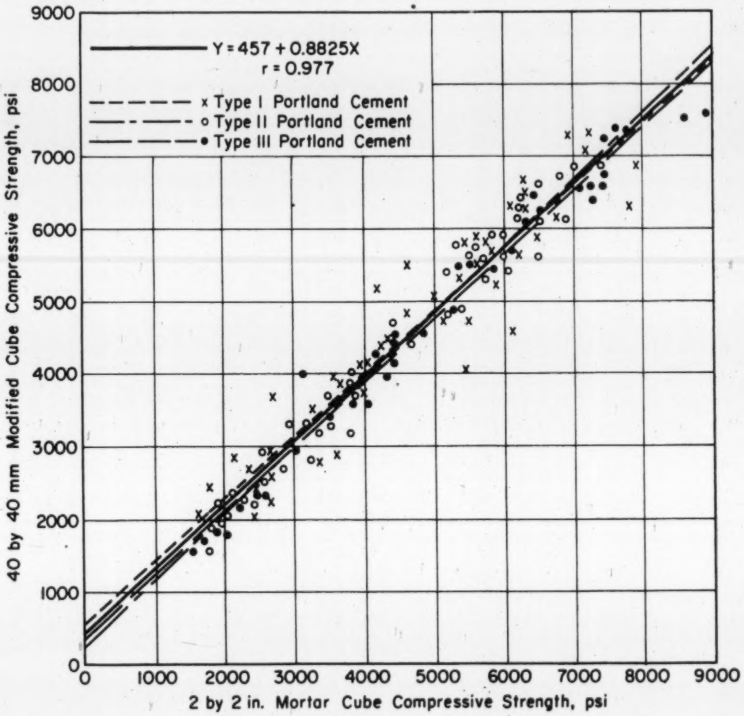

FIG. 2.—Line of Regression of Modified Cube Compressive Strength on Mortar Cube Compressive Strength.

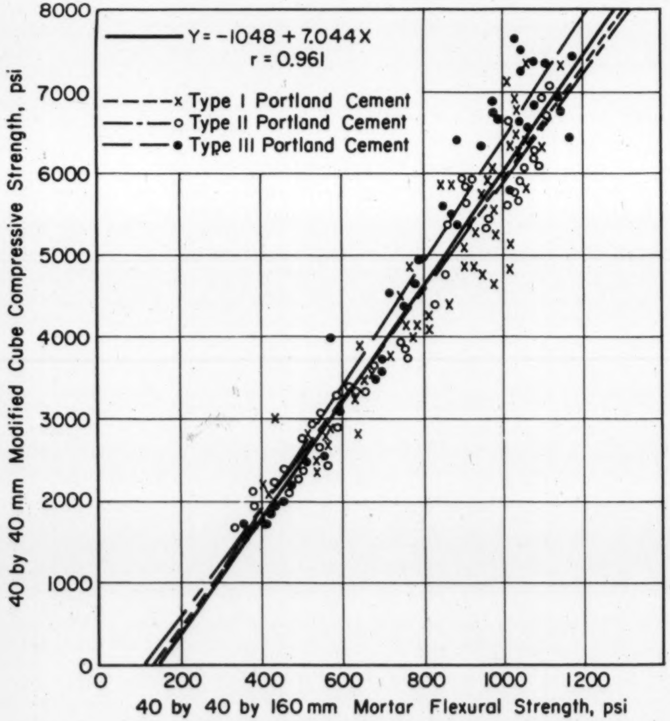

FIG. 3.—Line of Regression of Modified Cube Compressive Strength on Mortar Flexural Strength.

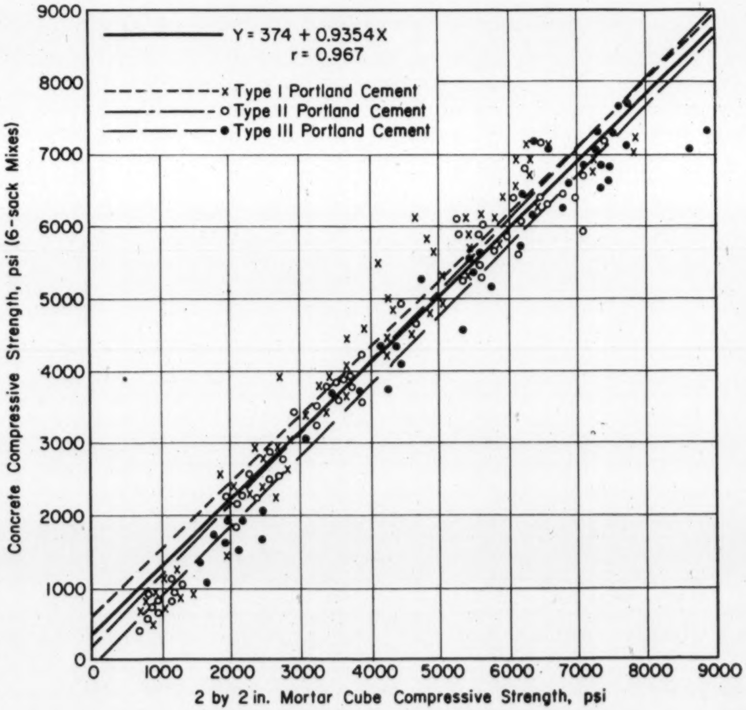

FIG. 4.—Line of Regression of Concrete Compressive Strength on Mortar Cube Compressive Strength.

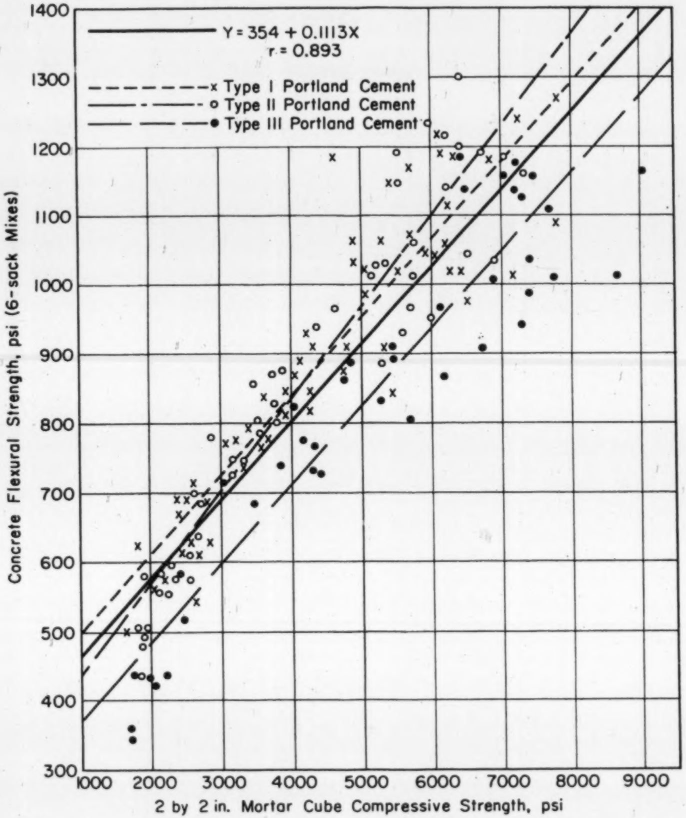

FIG. 5.—Line of Regression of Concrete Flexural Strength on Mortar Cube Compressive Strength.

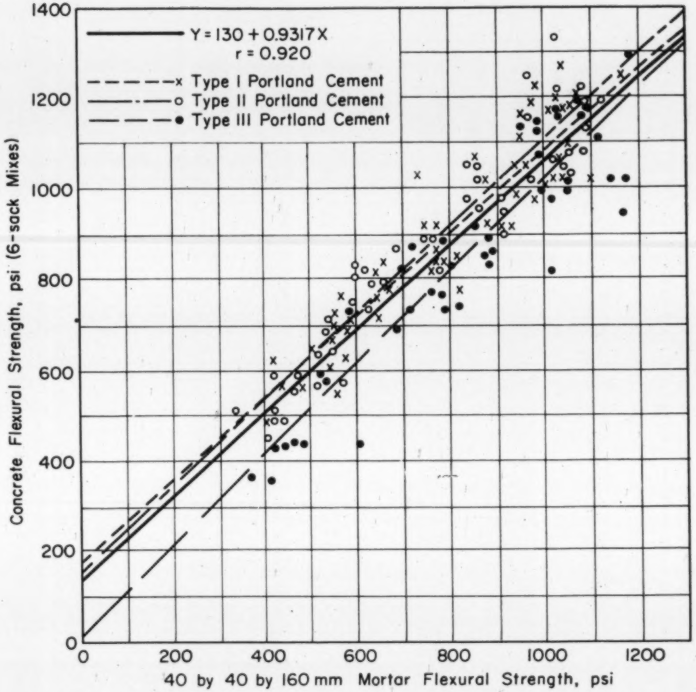

FIG. 6.—Line of Regression of Concrete Flexural Strength on Mortar Flexural Strength.

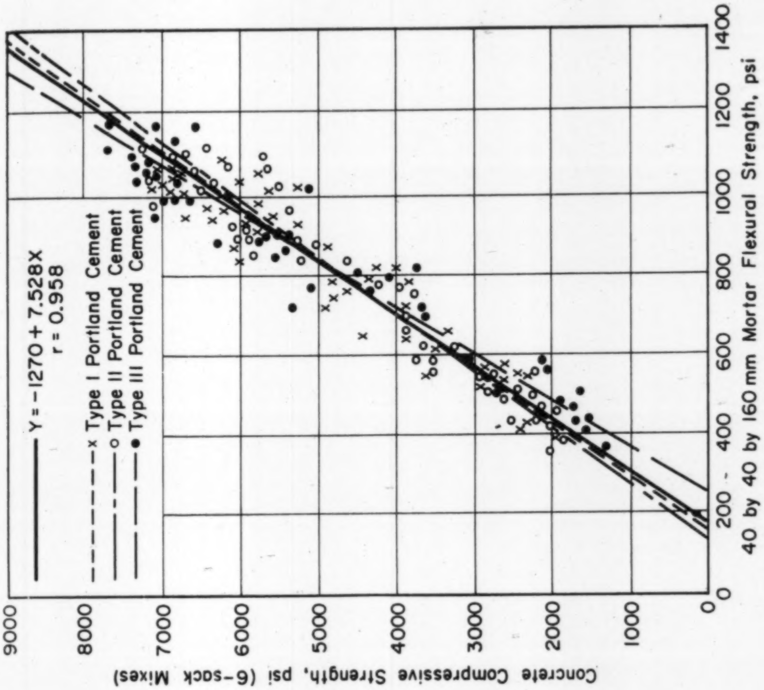

FIG. 8.—Line of Regression of Concrete Compressive Strength on Mortar Flexural Strength.

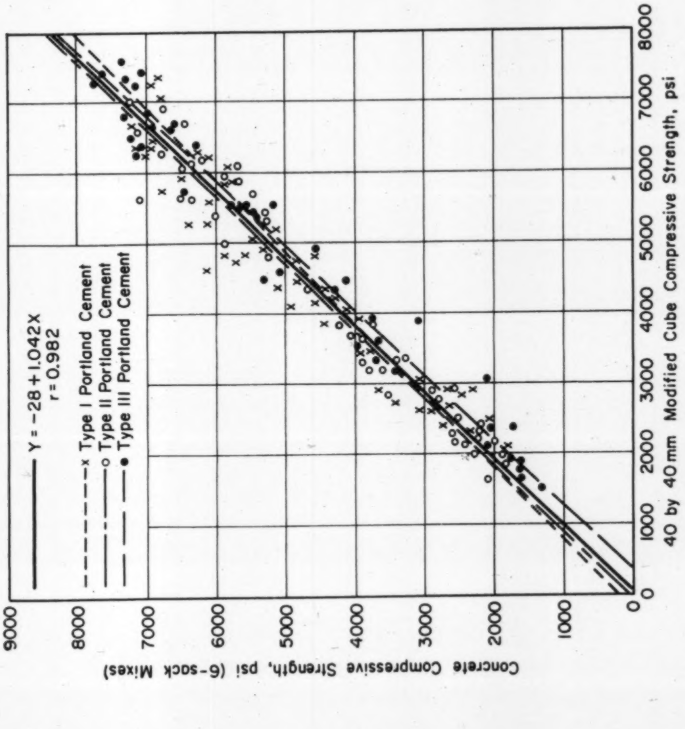

FIG. 7.—Line of Regression of Concrete Compressive Strength on Modified Cube Compressive Strength.

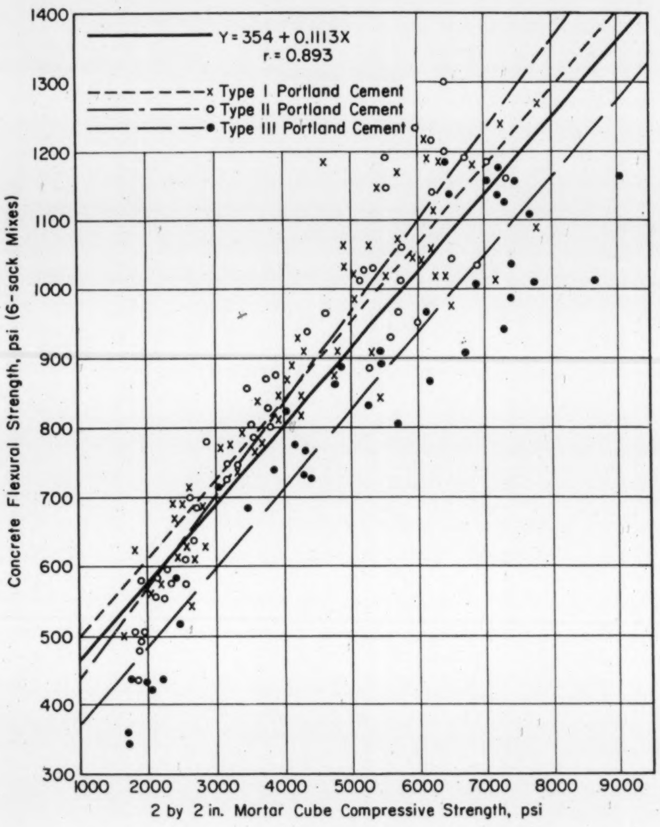

FIG. 5.—Line of Regression of Concrete Flexural Strength on Mortar Cube Compressive Strength.

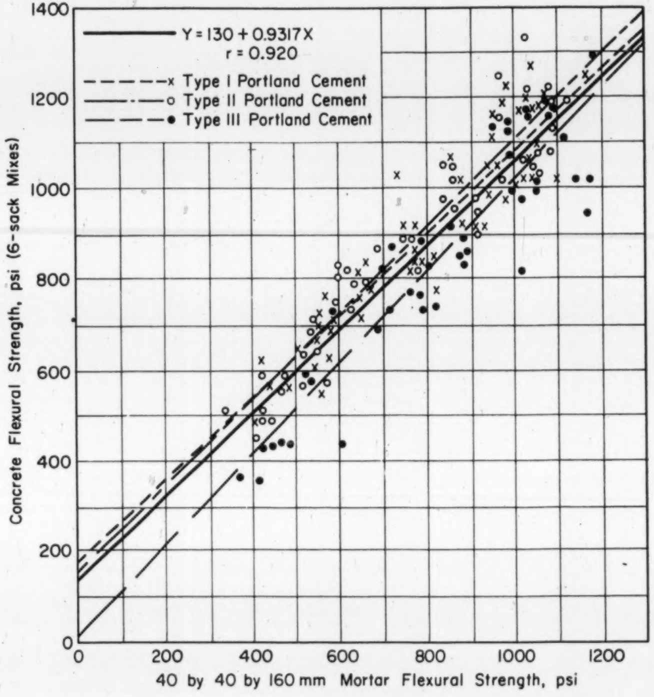

FIG. 6.—Line of Regression of Concrete Flexural Strength on Mortar Flexural Strength.

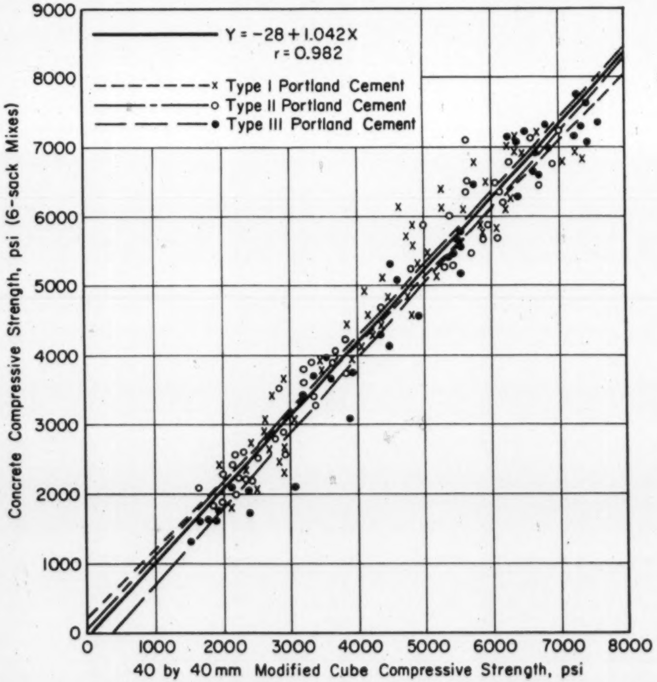

FIG. 7.—Line of Regression of Concrete Compressive Strength on Modified Cube Compressive Strength.

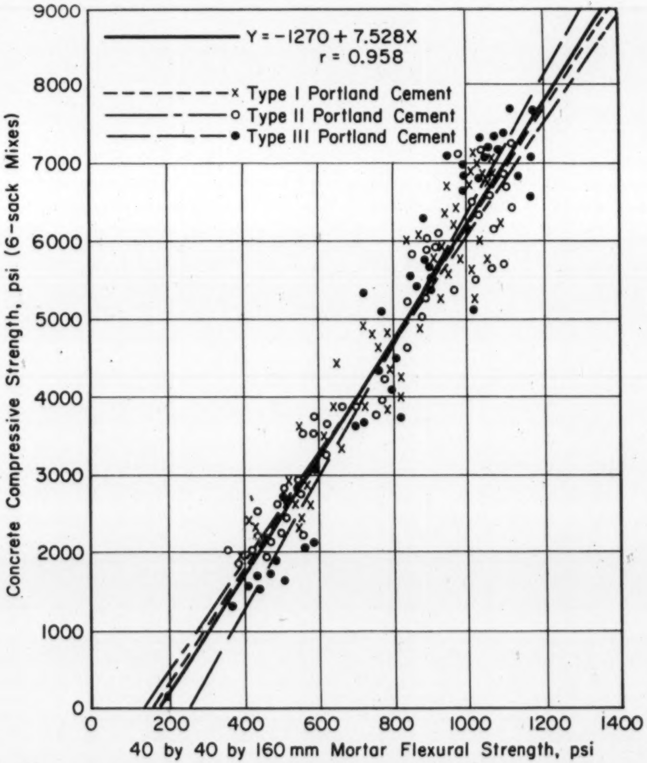

FIG. 8.—Line of Regression of Concrete Compressive Strength on Mortar Flexural Strength.

coefficient is one, then a perfect correlation exists and all values would fall on the regression line. Conversely, if the *r* value is 0, no correlation exists and no direct relationship between the two test results would be indicated.

Comparing the correlation coefficients for these regression lines with tabulated values from statistical handbooks for the number of degrees of freedom ($n - 2$), it was determined that these twelve relationships are highly significant. Equation 5-C, with 155 degrees of freedom, has the lowest correlation coefficient, 0.893. From the tabulated values of correlation coefficients, this value could be as low as 0.205 and meet the requirements of being significant at the 99 per cent level.

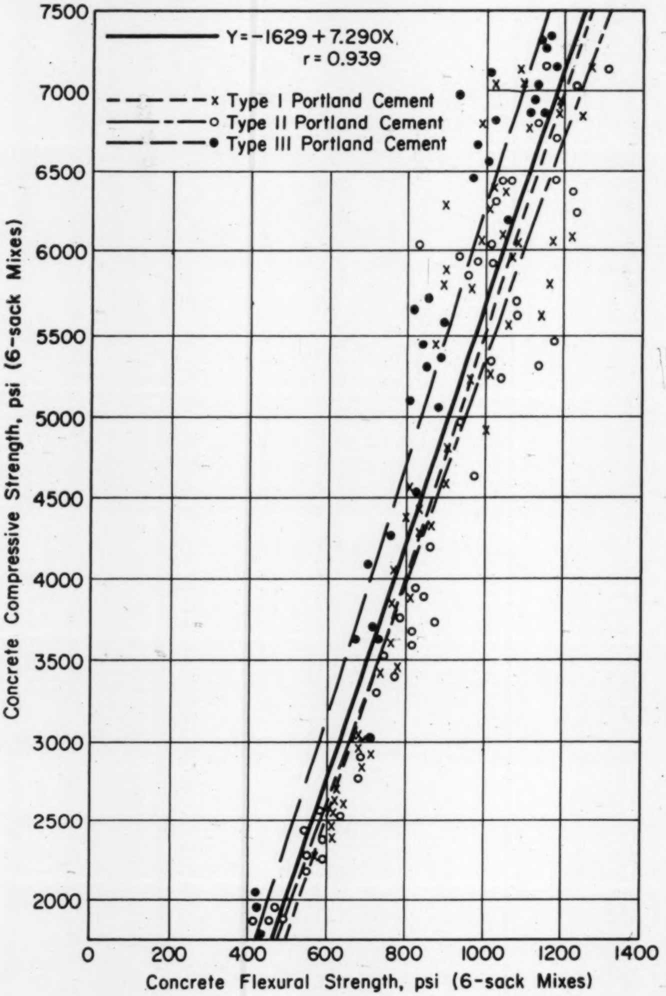

FIG. 9.—Line of Regression of Concrete Compressive Strength (6-Sack Mixes) on Concrete Flexural Strength (6-Sack Mixes).

Figures 1 to 12 illustrate the strength

data and the regression line for each cement type and with the three cement types combined. The equation shown on each of these figures is for the combined cements.

presented in Table IV, and the regression lines are plotted on each of the figures.

The precision of the regression equations was measured by computing the standard error of estimate. This value

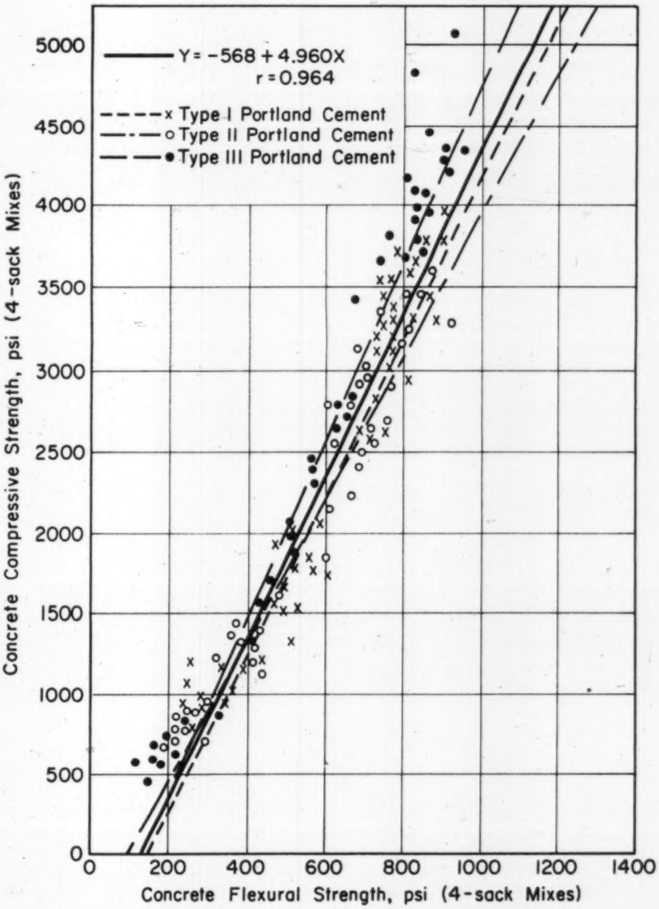

FIG. 10.—Line of Regression of Concrete Compressive Strength (4-Sack Mixes) on Concrete Flexural Strength (4-Sack Mixes).

Since the correlation coefficients for the equations using the combined cement types were highly significant and the similarity of the regression lines was noted when plotted on the accompanying figures, an analysis of the equations for each cement type was not made. The equations for each type of cement are for each of the equations as shown in column 2 of Table V indicates that two thirds of the data will fall within plus and minus the standard error when applied to the *Y* values or dependent variables for each of the equations.

The standard error of estimate, as applied to a regression equation, is a

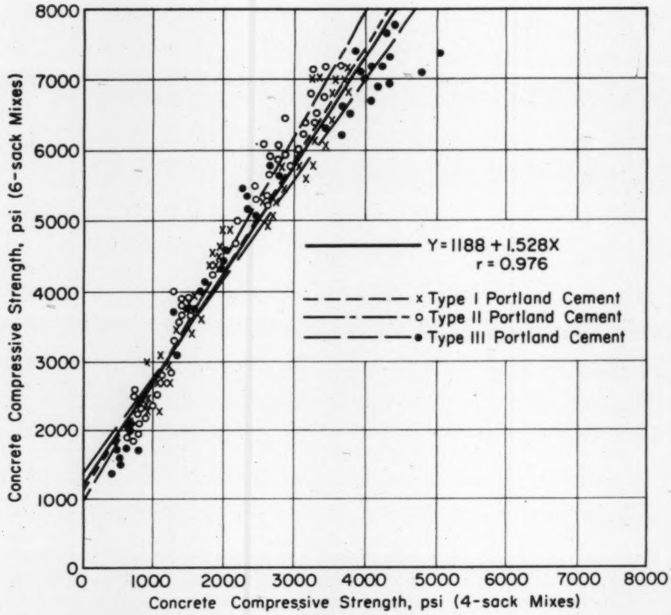

FIG. 11.—Line of Regression of Concrete Compressive Strength (6-Sack Mixes) on Concrete Compressive Strength (4-Sack Mixes).

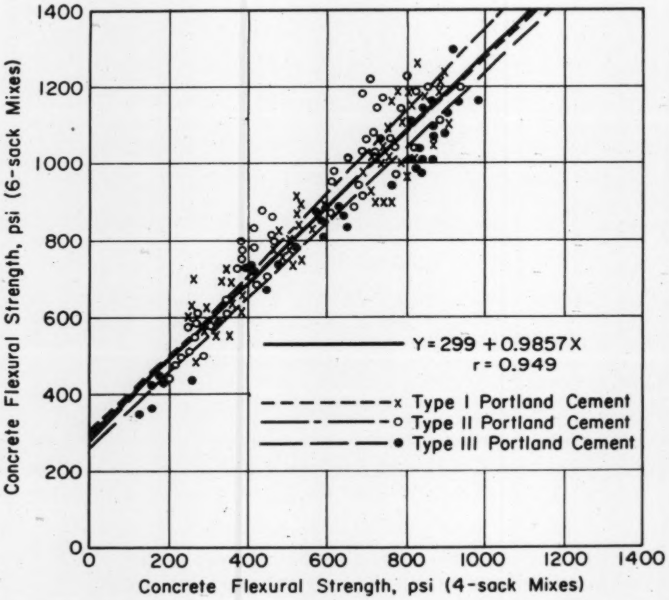

FIG. 12.—Line of Regression of Concrete Flexural Strength (6-Sack Mixes) on Concrete Flexural Strength (4-Sack Mixes).

term similar to the standard deviation for grouped data. The American Concrete Inst. (6) has shown that one can expect a coefficient of variation of laboratory test data to be as high as 7 per cent for good test work. In other words, if the average strength of test results was 4000 psi, a variation or standard deviation of ±400 psi would not be unreasonable. Since a regression equation involves two sets of data, we could expect a coefficient of variation of 7 per cent for each set or approximately 10 per cent variation for the regression equation. This is approximately the case since, for example, by use of equation 4-C we can predict the compressive strength of concretes from the mortar cube strength and expect a ±460-psi variation at 4000 psi.

TABLE V.—PRECISION OF REGRESSION EQUATIONS.

Equation Number (from Table IV)	Standard Error of Estimate, psi	Ratio of Y Value to X Value	
		Low Strength	High Strength
1-C	600	4.00	6.22
2-C	350	1.11	0.95
3-C	480	4.43	6.00
4-C	460	1.12	0.99
5-C	110	0.29	0.16
6-C	90	1.26	1.06
7-C	410	1.03	1.04
8-C	500	4.35	6.26
9-C	600	4.58	5.93
10-C	310	2.12	4.25
11-C	400	2.72	1.83
12-C	70	2.48	1.36

Columns 3 and 4 of Table V present the ratio of the Y (dependent variable) to the X (independent variable) for each of the related strengths at a low and high strength level. The ratio of compressive strengths to flexural strengths is approximately 4 for low strengths and 6 for high strengths. The ratio of compressive-to-compressive and flexural-to-flexural strengths is approximately 1, regardless of the strength level. The exception to these ratios is in cases where the 4-sack concretes are involved.

The ratio of the 4-sack concrete compressive strength to flexural strength is approximately 2 for low strengths and 4 for the high-strength level. The ratio of 6-sack concrete strengths to 4-sack concrete strengths is approximately 2.5 for low strengths and 1.5 for high strengths.

It should be pointed out that these strength relationships were developed from controlled laboratory concretes and mortars. Similar equations would be found by using data from other concrete investigations. Different aggregate sources, gradations, concrete slumps, and other factors which influence strength might produce different relationships.

CONCLUSIONS

1. This project illustrates that excellent correlations exist between various strength test results and that once the equations are determined, they can be used for computing other strength results. The following strength results can be determined within the expected limits of repeatability of laboratory testing:

   (*a*) Mortar flexural strength from mortar compressive strength,
   (*b*) Modified mortar cube compressive strength from standard 2-in. cube compressive strength,
   (*c*) Concrete flexural strength from concrete compressive strength, and
   (*d*) Concrete strengths for various cement contents when other mix design factors remain the same.

2. The development of equations for various cement types is not required, since the regression equations for the combination of all cements are not significantly different from those for the individual types.

Acknowledgment:

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STRAIN DISTRIBUTION IN COMPRESSIVELY LOADED CONCRETE SPECIMENS*

By John R. Keeton[1]

Synopsis

Experimental short-time loading tests were made on portland-cement concrete cylindrical specimens to reveal relative strain-sensing capabilities of mechanical and resistance strain gages. In addition, end-to-end strains were measured with resistance-type cantilever reeds.

Determination of end-to-end strain distribution by using mechanical and resistance gages as well as the newest photoelastic technique revealed extreme variations in strains both inside the concrete and along the outer surface of the cylindrical specimens.

Comparative tests indicated that strain measurements made with devices containing electrical resistance strain-sensing elements are more reliable because they reveal little effects of test variables.

Tests with the photoelastic coating technique indicated that contours of maximum shear strains follow no set rule regarding mortar and larger aggregate. The concrete acts like a rigid skeleton in which the strains are distributed indiscriminately around, across, and among pieces of large aggregates and the mortar which surrounds them.

In a study of concrete creep and shrinkage characteristics[2] considerable differences were observed between longitudinal compressive strains measured in the central portions of cylindrical specimens and those measured end-to-end in the same specimens. Central portion strain measurements were made with mechanical strain gages using cast-in-place metallic screws as reference points. The end-to-end measurements were made with cantilever deflection-type reeds instrumented with electrical-resistance strain gages. It was deemed advisable to determine by experimental investigation whether the differences between the two strain values could be attributed to the measuring methods or whether an anomaly had been disclosed.

Accordingly, experimental compressive static loading tests (not time-dependent) were conducted to disclose the true relationship between these methods of measurement and to reveal the distribution of strains throughout the length of cylindrical concrete specimens. For comparative purposes, strain values are reported in microinches per inch unless indicated otherwise.

The test specimens were concrete cylinders 6 in. in diameter and 18 in. long. The cylinders were cast vertically, and the top surfaces were hand finished with a steel trowel. The concrete was

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[1] Senior Project Engineer, Materials Division, U. S. Naval Civil Engineering Laboratory, Port Hueneme, Calif.

[2] John R. Keeton, "Time Dependent Deformations of Plain Concrete," *Highway Research Board Proceedings*, Vol. 39, pp. 310–335 (1960).

made with type III portland cement, and the maximum size aggregate was $\frac{3}{4}$ in. For 1 cu ft, the quantities by weight of the ingredients were as follows: cement, 28.20 lb; fine aggregate, 46.84 lb; coarse aggregate, 58.48 lb. Water-cement ratio was 5.16 gal per sack. The specimens were cured for 24 days in fog and subsequently for about six months in 20 per cent relative humidity (73 F). Compressive strength at time of testing was about 6500 psi. For loading the test specimens, a 400,000-lb. hydraulic testing machine was utilized in which the load was applied by upward movement of the platen.

INSTRUMENTATION

Mechanical Strain Gages:

Mechanical fulcrum-plate strain gages were used to measure the deformation between two metal gage-points embedded $\frac{7}{16}$ in. in the concrete (inserts) or between gage points affixed to the outer surface of the concrete (outserts). Deformation measurement for a given load was converted to strain over the length of the gage. Two longitudinal gage lines were placed diametrically opposite one another on each specimen, and strains reported represent average values for the two gage lines. Accuracy of the mechanical strain gages was verified with a specially-made micrometer device.

Resistance Strain Gages:

To enable better definition of compressive strain distribution, bonded electrical resistance strain gages of various lengths were cemented to the outer surface of the concrete on diametrically opposed longitudinal gage lines. The two diametrically opposite gages for each position were connected in series in the measuring arm of a Wheatstone bridge to obtain the average strain of the two sides of the specimen. The gage lines of the resistance strain gages were placed 90 deg radially from the mechanical strain gage lines. Initial and final strain readings were made with an electronic strain indicator through a switching and balancing unit.

Cantilever Reeds (End-to-End Measurement):

Two cantilever deflection reeds were mounted diametrically opposite one another to measure the end-to-end deformation of the loaded specimen.[2] Installation of the reeds is illustrated in Fig. 1. The reed block was anchored to the lower bearing plate and the brass rod holding the reed was anchored to the upper plate. Two resistance strain gages were placed on each reed, and the four gages were connected in a Wheatstone bridge to provide complete temperature compensation and to indicate, by prior calibration, the average end-to-end strain of the specimen. The bridge resistance readings were made on an electronic strain indicator, and strains were computed by using a calibration factor.

Photoelastic Coating Method of Strain Measurement:

In recent years, a variation of the photoelastic method of stress analysis known as the photoelastic coating method has been developed.[3] The photoelastic coating technique differs from conventional photoelasticity[4] in that the strain in the structure itself can be observed without resorting to a model composed of plastic, bakelite, or similar substance. A special transparent plastic is bonded directly to the surface of the structure to be studied. Upon being strained, the plastic becomes birefringent, and, when viewed in a field of polarized light through a reflection polariscope, black and colored fringe patterns are observed which are directly proportional to the intensity of strain. Thus the complete pattern of mechanical strains in a loaded structure can be observed.

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[3] Felix Zandman, "PhotoStress—Principles and Applications," Booklet published by Tatnall Measuring Systems Co., Phoenixville, Pa., pp. 1–16.

[4] M. Hetenyi, Ed., "Handbook of Experimental Stress Analysis," John Wiley and Sons, Inc., New York, N. Y., pp. 74–76; 828–976 (1950).

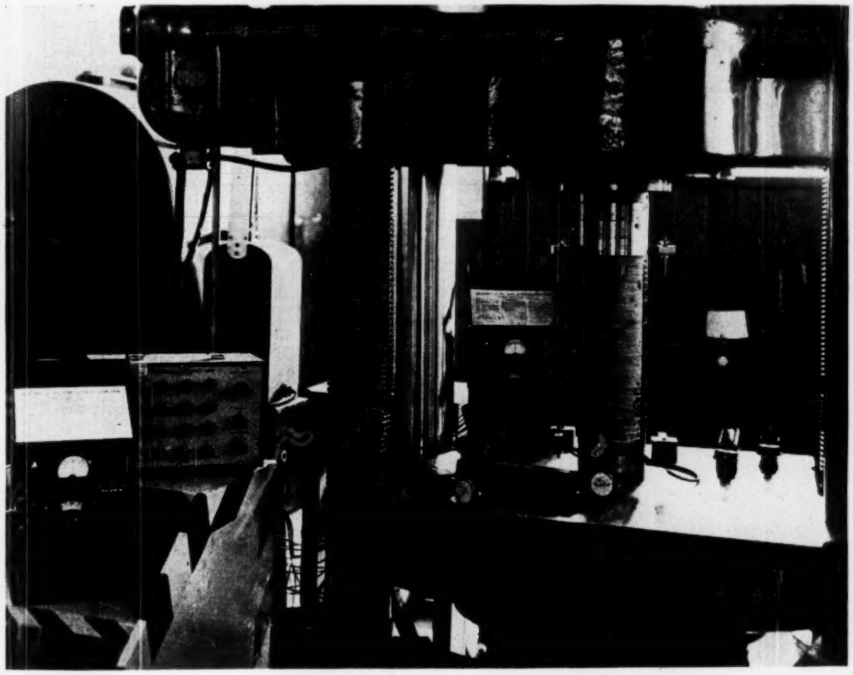


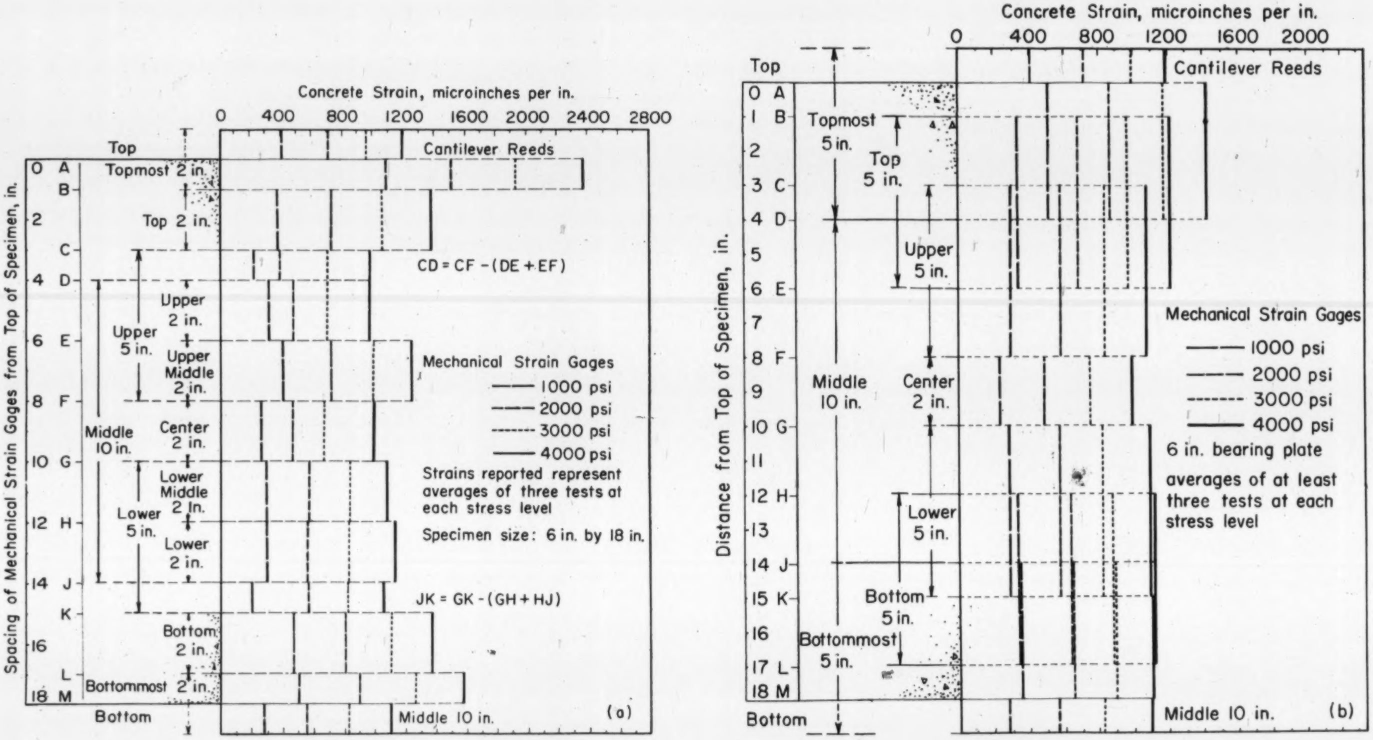
FIG. 1.—Test Installation to Determine Strain Distribution.

Experimental Testing Program:

Loading tests were conducted using a 6 by 18-in. concrete specimen on which strain measurements were made simultaneously with mechanical strain gages, resistance strain gages, and cantilever reeds. The entire test installation is pictured in Fig. 1. Mechanical strain gage inserts were placed in 2-in., 5-in., and 10-in. gage lengths, as indicated in Fig. 2, to enable overlapping measurements using different gages. Metal disks to be used as outsert points were cemented to the concrete surface adjacent to some of the inserts.

Resistance strain gages with gage lengths of $\frac{1}{8}$ in., $\frac{1}{2}$ in., $\frac{13}{16}$ in., 1 in., and 6 in. were cemented to the concrete surface in separate diametrically opposite longitudinal gage lines. The gages were placed in such a manner that the longer gages overlapped the shorter gages. The $\frac{1}{8}$-in. gages were positioned as close to the top and bottom surfaces as physically possible. The locations and spacings of the various resistance gages are indicated on the illustrations presented with each of the tests (Fig. 3).

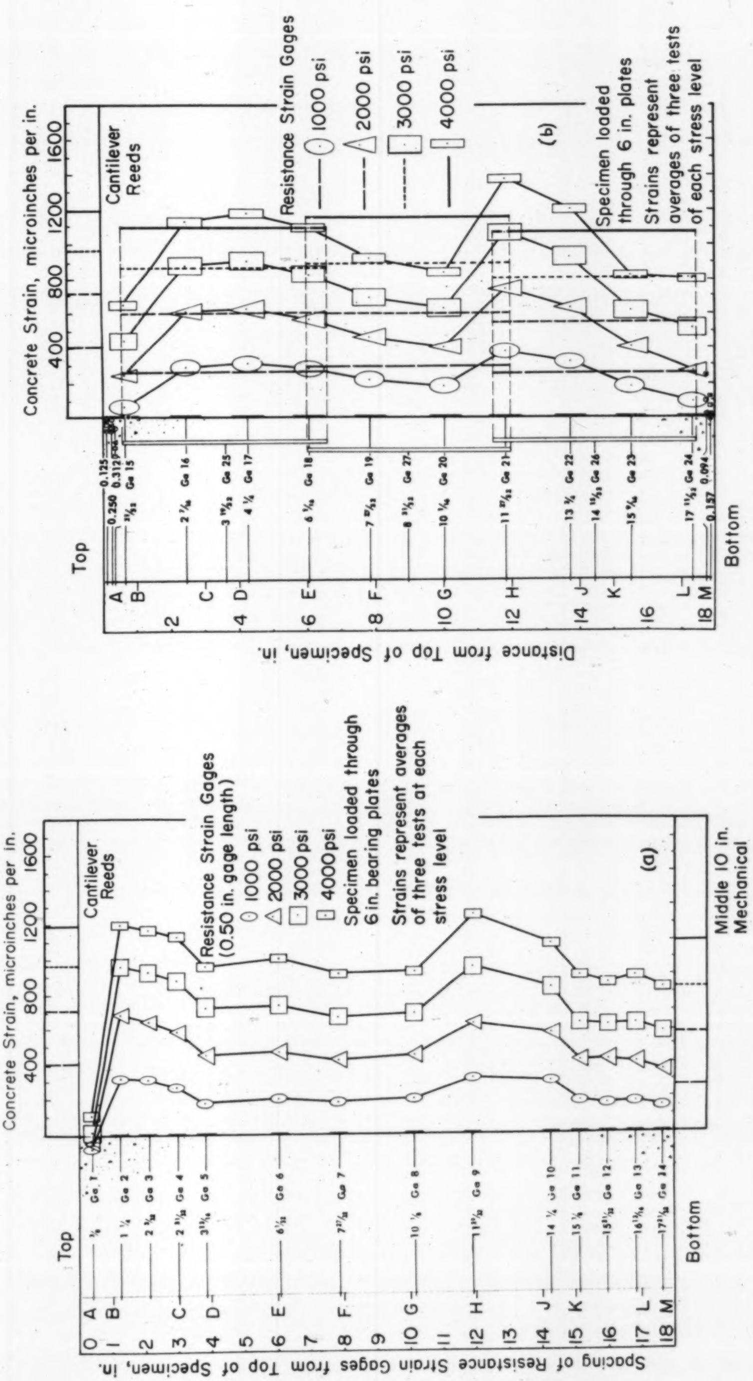
The specimen was loaded between 6-in. diameter steel bearing plates 1 in. thick, and the hand-finished end of the specimen was at the top. A seating load of 100 psi was used to obtain the initial or "no load" readings. All measurements were



(*a*) Measurements with 2-in. and 5-in. gages.

(*b*) Measurements with 5-in. and 10-in. gages.

FIG. 2.—Strain Distribution by Mechanical Strain Gages.



(a) ½-in. gage.

(b) ⅛-in., $\frac{13}{16}$-in., 1-in., and 6-in. gages.

FIG. 3.—Strain Distribution with Resistance Gages.

TABLE I.—SUMMARY OF RELATIONSHIPS BETWEEN MECHANICAL AND CANTILEVER MEASUREMENTS.

(Strains are in microinches per inch.)

Applied Stress, psi	Average End-to-End Strain				Ratio: Mechanical Gages to Cantilever Reeds		
	Mechanical Strain Gages[a]			Cantilever Reeds			
	2 in.	5–2–5 in.	5–10–5 in.		2 in.	5–2–5 in.	5–10–5 in.
1000....	366	351	343	411	0.89	0.85	0.83
2000....	680	637	627	714	0.95	0.89	0.88
3000....	965	904	902	965	1.00	0.94	0.93
4000....	1240	1159	1169	1194	1.04	0.97	0.98

[a] Insert gage points.

TABLE II.—COMPARISONS OF INSERT AND OUTSERT MEASUREMENTS (MECHANICAL).

(NOTE.—All strains are in microinches per inch.)

Mechanical Measurement	1000 psi			2000 psi			3000 psi			4000 psi		
	Insert	Outsert	Ratio[a]	Insert	Outsert	Ratio[a]	Insert	Outsert	Ratio[a]	Insert	Outsert	Ratio[a]
Topmost, 2 in., (AB)..............	1085	796	0.73	1527	1372	0.90	1894	1770	0.93	2370	2084	0.88
Top, 2 in., (BC)..................	380	450	1.18	730	755	1.03	1075	1035	0.96	1379	1300	0.94
Topmost, 5 in., (AD)..............	515	515	1.00	877	862	0.98	1173	1190	1.01	1426	1529	1.07
Middle, 10 in., (DJ)..............	276	269	0.97	556	550	0.99	825	821	0.99	1096	1090	0.99
Bottom, 2 in., (KL)...............	455	370	0.81	810	670	0.83	1110	950	0.86	1370	1210	0.88
Bottommost, 2 in., (LM)...........	486	350	0.72	930	725	0.78	1300	1110	0.85	1647	1414	0.86

[a] Ratio is outsert value to insert value.

made simultaneously and required about 90 sec for each loading.

Strain Distribution by Mechanical Gages:

Average strains for at least 3 tests each at stresses of 1000, 2000, 3000, and 4000 psi are presented in Fig. 2 for comparisons between mechanical strain gages and cantilever reeds.

For clarity, the average measurement for the mechanical gage lengths is depicted in Figs. 2(*a*) and 2(*b*) as uniform over the entire gage length. The topmost and bottommost gage lengths included 1 in. of the respective steel bearing plates. The steel strain has been deducted; the values reported are concrete strains. In Fig. 2(*a*) strain distribution is pictured as determined by the 2-in. and 5-in. mechanical insert measurements. The strain is fairly uniform in the central portion but high strains are seen in the ends. Figure 2(*b*) depicts strain as measured with the 5-in. and 2-in. mechanical gages (insert gage points) in locations as shown.

Pertinent relationships between mechanical and cantilever measurements are summarized in Table I. The center 2-in. value was utlized in computation of the end-to-end strain with the 5-in. measurements. Referring to the mechanical gage measurements in terms of concrete segments as designated in Fig. 2, an example of the computation of the end-to-end strain for the 5–2–5 in. method is as follows: *AD* plus *CF* minus *CD* plus *FG* plus *GK* minus *JK* plus *JM*. The strains in segments *CD* and *JK* were determined by utilizing the upper and lower 5-in. measurements (*CF* and *GK*) which overlapped the 2-in. segments *DE-EF* and *GH-HJ*. Generally speaking, end-to-end compilations using the 2-in. measurements agreed most closely with the cantilever values, probably because one of the inserts was common to the measurement above and below, thus cancelling any effect of bulging of the concrete or differential strains along the embedded insert. The greatest difference is observed with the 5–10–5 in. computation at 1000 psi (−17 per cent).

TABLE III.—RESISTANCE STRAIN GAGE AND CANTILEVER DATA.

(NOTE.—Strains are in microinches per inch.)

Gage Number	Gage Length, in.	Gage Span From Top of Specimen, in.	Applied Stress			
			1000 psi	2000 psi	3000 psi	4000 psi
1	½	0.12 to 0.62	79 *T*[a]	52 *T*	20	123
2	½	1.00 to 1.50	330	680	965	1204
3	½	1.84 to 2.34	314	642	925	1181
4	½	2.72 to 3.22	266	564	845	1132
5	½	3.56 to 4.06	180	436	686	945
6	½	5.78 to 6.28	220	484	745	999
7	½	7.59 to 8.09	177	424	666	914
8	½	9.88 to 10.38	220	452	689	926
9	½	11.66 to 12.16	330	652	943	1228
10	½	14.00 to 14.50	301	560	829	1090
11	½	14.88 to 15.38	200	424	645	901
12	½	15.72 to 16.22	175	396	617	854
13	½	16.56 to 17.06	160	384	632	900
14	½	17.41 to 17.91	95	210	375	633
15	13/16	0.25 to 1.06	70	250	449	655
16	1	1.94 to 2.94	288	608	890	1130
17	1	3.75 to 4.75	310	624	897	1179
18	1	5.56 to 6.56	279	565	832	1094
19	1	7.34 to 8.34	223	468	695	923
20	1	9.56 to 10.56	195	410	625	846
21	1	11.34 to 12.34	382	744	1072	1380
22	1	13.25 to 14.25	318	636	930	1205
23	1	15.06 to 16.06	190	400	614	823
24	13/16	16.94 to 17.75	100	275	524	812
25	6	0.59 to 6.59	275	580	872	1140
26	6	11.47 to 17.47	252	525	802	1076
27	6	5.97 to 11.97	295	587	850	1113
28	⅛	0.06 to 0.18	3 *T*	35 *T*	68 *T*	105 *T*
29	⅛	0.18 to 0.30	16 *T*	45 *T*	71 *T*	82 *T*
30	⅛	0.25 to 0.37	115 *T*	213 *T*	243 *T*	230 *T*
31	⅛	17.78 to 17.90	8	26	101	246
32	⅛	17.84 to 17.96	5 *T*	22	68	123
Cantilever Reeds		0.00 to 18.00	411	714	965	1194

[a] Tensile strains designated by "*T*."

Outsert disks were placed adjacent to inserts at several locations to enable direct comparisons between mechanical gage measurements over the same segments. Results are presented in Table II.

Comparisons of insert and outsert values for the topmost 5 in. and middle 10-in. measurements reveal insignificant differences, with a maximum of 7 per cent. Outsert values are predominantly lower than the insert values. Most significant differences occur with 2-in. gages. The range of ratios of outsert to insert measurements is −28 per cent to +18 per cent. It is believed that these differences between outsert and insert mechanical measurements reflect the non-uniformity of strain distribution in the specimen. The metal screw serving as an insert (embedded $\frac{7}{16}$ in.) is subjected to different strains along its length which, when combined with bulging of the concrete, results in some as yet undisclosed manifestation upon the free end of the screw. As stated previously, when each insert is common to two mechanical measurements, this effect is cancelled if the average of the two readings is utilized.

Strain Distribution by Resistance Gages:

The averages of at least three tests each at stresses of 1000, 2000, 3000, and 4000 psi are indicated for the resistance gages in Table III and in Fig. 3.

Strains as measured with ½-in. resistance gages are presented in Fig. 3(*a*). Tensile strains are observed in gage 1 at the top. Reasons for the presence of tensile strains are discussed later in the paper. Strain distribution as determined by ⅛-in., $\frac{13}{16}$-in., 1-in., and 6-in. resistance gages is illustrated in Fig. 3(*b*). Tensile strains are indicated in gages 28, 29, 30 and 32 (⅛-in.) near the top and bottom surfaces. It is seen that except for gages 15 and 24 ($\frac{13}{16}$ in.), near the top and bottom surfaces, the 6-in. gage measurements appear to be approximate averages of the measurements with the 1-in. gages. Strains in gages 15 and 24 reflect the tendency toward tensile strains near the top and bottom surfaces. As expected, non-uniformity in strain distribution is virtually undetected in measurements made with 6-in. gages. Comparisons between ½-in. and 6-in. gages and between 1-in. and 6-in. gages over the central 6 in. of the specimen (gage 27) are summarized in Table IV. Strains are in microinches per inch. Values for the ½-in. and 1-in. gages are averages for the gages spanning the same segment of concrete as gage 27 (6 in.).

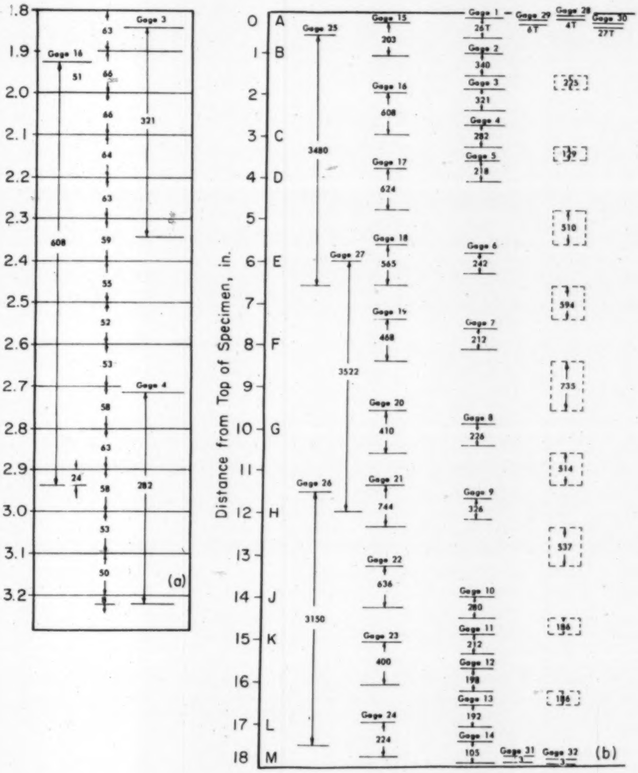
TABLE IV.—COMPARISONS BETWEEN GAGES OVER THE CENTRAL 6 IN. OF GAGE 27.

(Strains are in microinches per inch.)

Applied Stress, psi	Segment Covered by Gage 27			Ratio of 6-in. Gage to	
	½-in. Gages	1-in. Gages	6-in. Gages	½-in. Gages	1-in. Gages
1000.....	204	250	295	1.45	1.18
2000.....	443	512	587	1.32	1.15
3000.....	678	759	850	1.25	1.12
4000.....	913	1004	1113	1.22	1.11

The use of several lengths of resistance strain gages in these tests has enabled a unique determination of strain distribution in the specimen from end-to-end. Each resistance gage reading was converted to total deformation in microinches by multiplying the strain by the gage length. The total deformation in each gage was then weighted against the corresponding value in another gage which covered at least partially the same segment of concrete. Since the longer gages overlapped many of the shorter gages, it was possible to determine the total deformation in segments of the concrete as short as 0.10 in. An example of the computation sheet is presented in Fig. 4(*a*), in which gage 16 (1 in.) overlaps gages 3 and 4 (½ in.). To develop the layout presented in Fig. 4(*a*), which indicates values for 2000 psi, the total deformation in each of the gages is divided by the number of 0.10-in. segments its length covers—5 for gages 3 and 4 and 10 for gage 16; thus the averages for 0.10-in. segments for gages 3, 4, and 16 were 64, 56, and 61, respectively. These averages for 0.10 in. reveal quite readily that in order to maintain a total deformation of 608 microinches for the 1-in. length, the deformations in the upper portion of the 1-in. gage length must be greater than those in the lower portion, and, by mathematical reasoning, that the middle segment values are smaller than either of the upper or lower portions.

Figure 4(*b*) illustrates the method used to estimate the strain distribution in segments not covered by ½ in. or 1-in. gages but overlapped by 6-in. gages. The boxes shown by dashed lines at the right of Fig. 4(*b*) indicate the segments of concrete along which it was necessary to estimate the strain distribution. Lengths of the estimated segments nearest the top and bottom were rather short (0.343 in.), thus simplifying the estimation. Although the dashed boxes nearer the center of the specimen extend over longer distances, the fluctuations of strain are not nearly so great in these central portions as in portions closer to the extremities.



(*a*) Representative portion of specimen.
(*b*) Complete specimen.

FIG. 4.—Chart for Estimating End-to-End Strain Distribution.

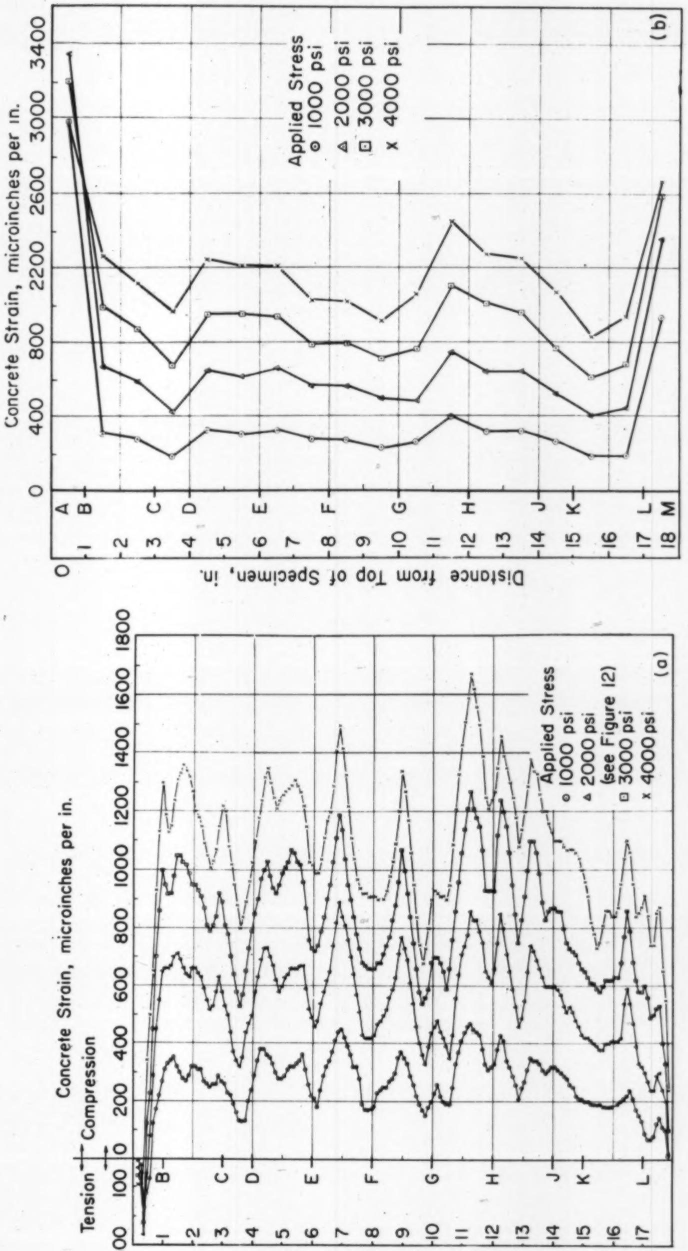
Examination of the top and bottom extremities of the layout of the specimen presented in Figure 4(*b*) reveal that determination of the end-to-end strain distribution necessitates an additional estimation—the amount of deformation in the top and bottom 0.10-in. segments 0.00 to 0.10 and 17.90 to 18.00 not covered by gages. This estimation can be made by using the cantilever reed measurements (end-to-end). Computation of the deformation in these extremities is presented in Table V. It should be stressed, as shown in Table V, that item 7 represents the total deformation *between bearing plates*, since the cantilevers are anchored to these plates. Item 8 indicates the total deformation to be divided between the top and bottom 0.10-in. segments. In order to distribute this deformation most reasonably between 0.00 to 0.10 and 17.90 to 18.00, the ratio of the topmost and bottommost 2-in. outsert values (mechanical measurements) were utilized. The outsert values were chosen instead of the insert values because the outsert disks were not influenced by differential strains acting inside the concrete and thus reflected only surface strains, as did the resistance gage measurements used in the above computations.

Figure 5(*a*) presents end-to-end strain distribution in 0.10-in. segments as determined by resistance gage measurements together with the strains obtained by the above computations. To separate strains for the various stresses, it was necessary to halve the scale used for other figures, thus tending to exaggerate the fluctuations. Extremely irregular strain distribution is observed from end-to-end, most likely reflecting variations in stress-strain response within the heterogeneous concrete mass. Strains in the top and bottom 0.10-in. portions, not shown in Fig. 5(*a*), are discussed later in the paper. The necessity of making strain measurements over relatively long gage lengths is vividly portrayed in Fig. 5(*a*).

TABLE V.—STRAIN COMPUTATIONS FOR 0.10-IN. EXTREMITIES OF SPECIMEN.
(NOTE.—Strains are total deformation in microinches; tensile strains designated by "*T*.")

Item	Designation of Segments Used in Computations, in. from top of specimen	Applied Stress			
		1000 psi	2000 psi	3000 psi	4000 psi
1......	0.10 to 0.59 (By gages and estimation)...........	39 *T*	31 *T*	0	48
2......	0.59 to 6.59 (gage 25)...............	1 650	3 480	5 232	6 840
3......	6.59 to 11.47 (Part of gage 27)......	1 428	2 829	4 082	5 335
4......	11.47 to 17.47 (gage 26).............	1 512	3 150	4 812	6 456
5......	17.47 to 17.90 (By gages and estimation)..........	43	85	155	258
6......	0.10 to 17.90 (Items 1 + 2 + 3 + 4 + 5).......	4 594	9 513	14 281	18 937
7......	0.00 to 18.00 (By cantilever reeds)...	7 398	12 852	17 370	21 492
8......	0.00 to 0.10 and 17.90 to 18.00 (Item 7 minus Item 6).............	2 804	3 339	3 089	2 555
9......	Topmost 2 in. (Mechanical strain gage outsert measurement).............	796	1 372	1 770	2 084
10......	Bottommost 2 in. (Mechanical strain gage outsert measurement)..........	350	725	1 110	1 414
11......	Ratio: Item 9 to Item 10...............	2.27	1.90	1.59	1.47
12......	Strain 0.00 to 0.10 (By weighting Item 8 as indicated by Item 11)..........	1 949	2 188	1 900	1 522
13......	Strain 17.90 to 18.00 (Item 8 minus Item 12).........................	855	1 151	1 189	1 033

Strains in microinches per inch for the topmost and bottommost portions of the specimen are presented in Table VI. In Table VI, the strains indicated as being present in the segments 0.0 to 0.10 and 17.90 to 18.00 are questionable unless it is recognized that the end-to-end deformations from which these strains were computed were measured between bearing plates. Actually, much of the strain in these 0.10-in. extremities occurred in the center portion of the contact area between the bearing plates and the concrete because of the convexity of the con-



(*a*) 0.10-in. segments.

(*b*) 1.0-in. segments.

FIG. 5.—Strain Distribution by Resistance Gages Shown in Segments.

TABLE VI.—STRAINS FOR TOP AND BOTTOM PORTIONS OF THE SPECIME

(Strains in microinches per inch.)

Segments Referred to Top of Specimen, in.	Applied Stress			
	1000 psi	2000 psi	3000 psi	4000 psi
0.0 to 0.10	19 490	21 880	19 000	15 220
0.10 to 0.20	10 T^a	30 T	60 T	90 T
0.20 to 0.30	20 T	50 T	70 T	90 T
0.30 to 0.40	170 T	260 T	220 T	180 T
0.40 to 0.50	120 T	50 T	120	340
17.50 to 17.60	140	300	530	870
17.60 to 17.70	110	240	400	650
17.70 to 17.80	100	200	330	550
17.80 to 17.90	10	20	100	240
17.90 to 18.00	8 550	11 510	11 890	10 330

[a] T = Tensile strains.

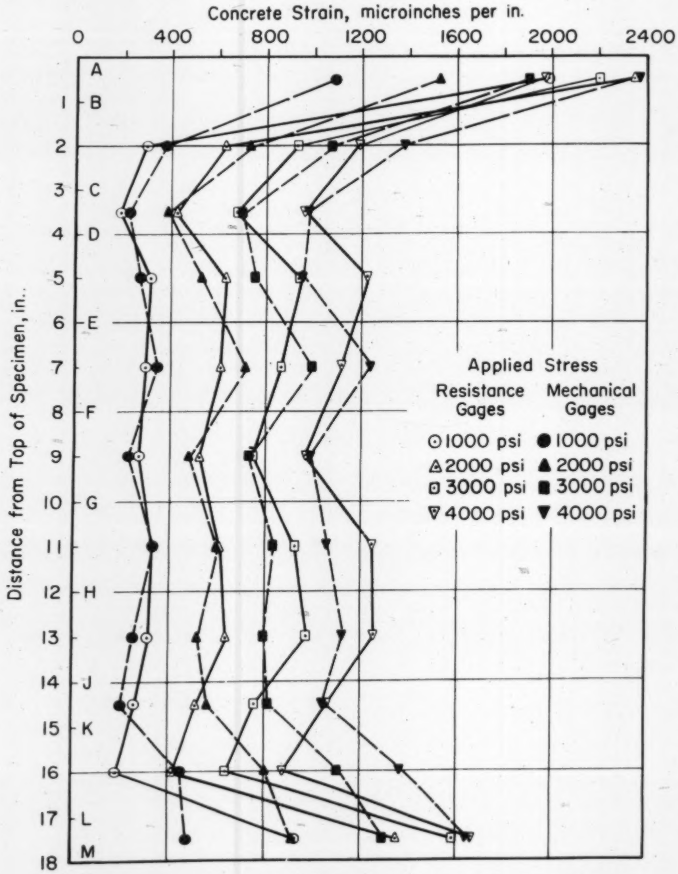


FIG. 6.—Strain Distribution by Mechanical and Resistance Gages.

crete top and bottom surfaces. The end conditions of the concrete are clarified in a subsequent section of the paper.

Figure 5(*b*) indicates strain distribution as determined by average strains for each 1-in. segment of the specimen. Comparison of Figs. 5(*a*) and 5(*b*) discloses considerable smoothing out of the fluctuations when the strain is computed over longer gage lengths.

TABLE VII.—RESISTANCE AND MECHANICAL GAGE MEASUREMENTS OF DESIGNATED SEGMENTS.

(NOTE.—Data presented are in microinches per inch.)

Segment Designation	1000 psi			2000 psi			3000 psi			4000 psi		
	Resistance	Mechanical	Ratio[a]	Resistance	Mechanical	Ratio[a]	Resistance	Mechanical	Ratio[a]	Resistance	Mechanical	Ratio[a]
AB (topmost 2 in.)	1988	1085	0.54	2351	1527	0.65	2201	1895	0.86	1971	2370	1.20
BC (top 2 in.)	296	380	1.28	630	730	1.16	929	1075	1.16	1190	1379	1.16
CD[b]	189	230	1.22	426	390	0.92	678	700	1.03	963	980	1.02
DE (upper 2 in.)	318	275	0.86	636	530	0.83	954	755	0.79	1229	955	0.78
EF (upper middle 2 in.)	302	350	1.16	615	720	1.17	866	1000	1.15	1120	1245	1.11
FG (center 2 in.)	258	230	0.89	530	485	0.92	755	745	0.99	966	995	1.03
GH (lower middle 2 in.)	334	340	1.02	618	610	0.99	932	840	0.90	1256	1066	0.85
HJ (lower 2 in.)	320	255	0.80	646	525	0.81	984	805	0.82	1263	1130	0.89
JK[b]	265	210	0.79	521	570	1.09	764	820	1.07	1069	1048	0.98
KL (bottom 2 in.)	190	455	2.39	422	810	1.92	648	1110	1.71	884	1370	1.55
LM (bottommost 2 in.)	934	486	0.52	1361	930	0.68	1592	1300	0.82	1672	1647	0.98
AD (topmost 5 in.)	692	515	0.74	1009	877	0.87	1184	1173	0.99	1329	1426	1.07
BE (top 5 in.)	283	335	1.18	591	682	1.15	889	972	1.09	1160	1214	1.05
CF (upper 5 in.)	286	296	1.03	586	578	0.99	863	842	0.98	1132	1076	0.95
GK (lower 5 in.)	315	280	0.89	610	568	0.93	919	822	0.89	1221	1088	0.89
HL (bottom 5 in.)	257	332	1.29	531	632	1.19	806	882	1.09	1073	1112	1.04
JM (bottommost 5 in.)	394	340	0.86	681	637	0.94	913	893	0.98	1128	1102	0.98
DJ (middle 10 in.)	306	276	0.90	609	556	0.91	898	825	0.92	1167	1096	0.94
AM (end-to-end)	411	366	0.89	714	680	0.95	965	965	1.00	1194	1240	1.04

[a] Ratios shown are mechanical measurements to resistance measurements.

[b] Mechanical strain in segments CD and JK computed as follows: CD = upper 5 in. minus upper middle 2 in. minus upper 2 in.; JK = lower 5 in. minus lower middle 2 in. minus lower 2 in.

Mechanical end-to-end strain determined by using 2 in. measurements.

Comparisons of Mechanical and Resistance Gage Measurements:

As indicated earlier, the *average* end-to-end strain values measured mechanically (using inserts) agree quite closely with the average strain determined by the cantilever reeds, the principal differences between the measurements lie in the distribution of the strains from end-to-end. These differences are emphasized in Fig. 6 and in Table VII which compare strains in designated segments. Considerable variations are observed between some of the mechanical and resistance measurements; as noted in previous tests, the variations tend to decrease as the stress increases. Most notable variations between mechanical and resistance gage measurements are observed in the shorter gage lengths, indicating that the use of short mechanical strain gages for measurement of the average concrete strain is rather questionable.[4] The magnitude of the differences between the two measurements in the topmost and bottommost extremities (AB and LM) again suggests undue influence upon the embedded inserts, namely, inserts at B and at L.

Tests Utilizing the Photoelastic Coating Method:

Definitions and assumptions (3):

The following definitions and assumptions were utilized.

$\sigma_1, \sigma_2, \sigma_3$ = the principal stresses acting on a three-dimensional body; in this study, $\sigma_3 = 0$ (two dimensional plane surface).

ϵ_1 = principal strain with the higher algebraic value,

ϵ_2 = principal strain with the lower algebraic value,

E = modulus of elasticity of concrete, and

μ = Poisson's ratio of concrete.

Isoclinics: Lines connecting points through which the inclinations of the principal strains are constant.

Isochromatics: Color fringes revealing the quantitative value of the difference between the principal strains, $\epsilon_1 - \epsilon_2$, or maximum shear strains.

Theoretical Developments: Since only surface strains are considered in this presentation, $\sigma_3 = 0$, and Hooke's equations for strains are as follows:

$$\epsilon_1 = \frac{\sigma_1}{E} - \mu\frac{\sigma_2}{E},$$

$$\epsilon_2 = \frac{\sigma_2}{E} - \mu\frac{\sigma_1}{E},$$

$$\text{then: } \epsilon_1 - \epsilon_2 = (\sigma_1 - \sigma_2)\left(\frac{1 + \mu}{E}\right).$$

also, by photoelastic determination: $\epsilon_1 - \epsilon_2$ = isochromatic color value in microinches per inch. At any free boundary, the stress normal to the free boundary is zero, and the direction and quantity of ϵ_2 can be determined, parallel to any free boundary. At the free bound-

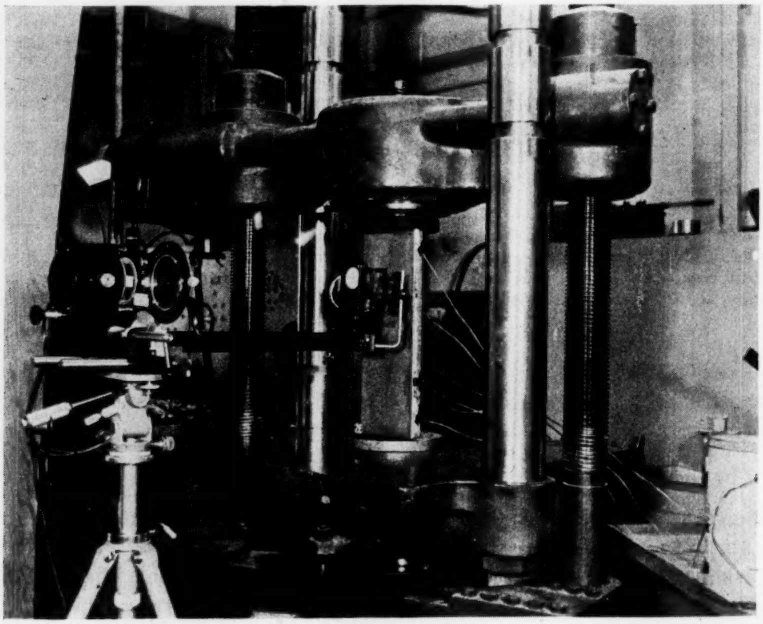


FIG. 7.—Test Installation for Photoelastic Coating Tests.

ary of the concrete: $\epsilon_2 = \dfrac{\epsilon_1 - \epsilon_2}{1 + \mu}$ and $\epsilon_1 = -\mu\epsilon_2$.

Isochromatic color patterns observed in the birefringent plastic through the reflection polariscope indicate differences between the principal strains $(\epsilon_1 - \epsilon_2)$, that is, maximum shear strains. Magnitude of the principal strain difference at any point is determined by differentiation among various colors and by the location of the point with respect to a fringe between red and blue or red and green (called a "tint of passage"). For the type and thickness of birefringent plastic used on the concrete, the first order fringe (tint of passage) represents a principal strain difference of 945 microinches per inch.

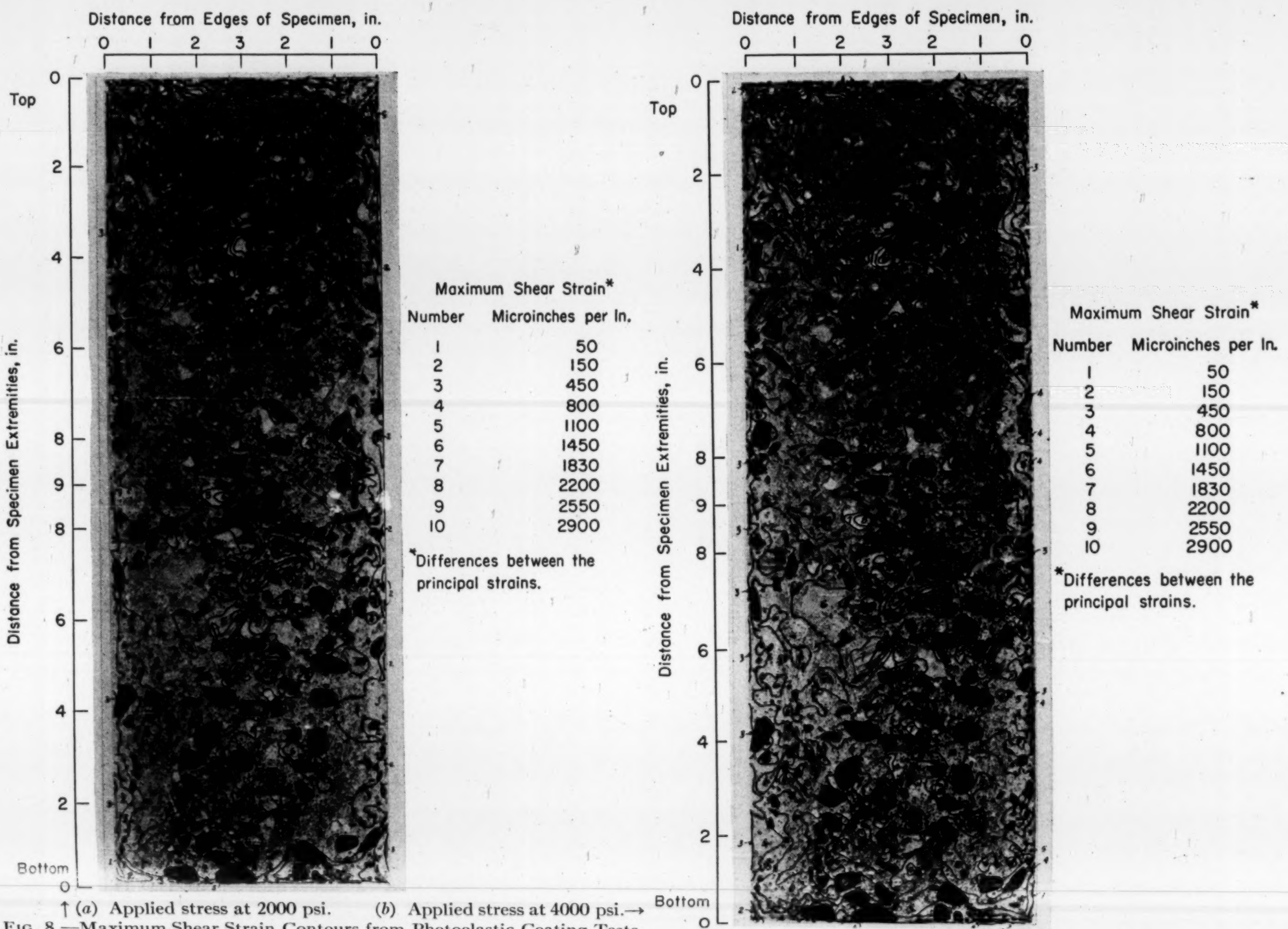
Quantitative determinations of the value of ϵ_1 and ϵ_2 are accomplished with the oblique incidence attachment to the reflection polariscope by making certain adjustments and settings; isoclinics—lines connecting points where the directions of principal strains are constant—can also be viewed through the reflection polariscope. A view of the reflection polariscope with oblique incidence attachment in place is presented in Fig. 7.

To provide a flat concrete surface upon which to install the plastic sheet, a 6 by 18-in. cylinder, companion to the one used in the tests reported above, was sliced in half longitudinally. The specimen with plastic sheet attached is shown in Fig. 7. As before, the specimen was loaded through steel plates on each end, and a spherically-seated loading head was used in the testing machine with the hand-finished end at the top.

Historically, measurements of concrete compressive strains for determination of mechanical properties have been made on the outer surface of the concrete. In actuality, the outer surface is not representative of the concrete per se but is a layer of mortar the thickness of which depends upon casting procedures and techniques. For practical purposes the strains obtained in this manner are generally accepted as representative of the strain response of the concrete mass. Longitudinal slicing of the specimen used in this study provided an interface with a new plane on which strain distribution could be determined. On the new plane the geographical orientation of aggregate and mortar is revealed as it actually existed upon being hardened into concrete. It is admitted that in the interior of an actual specimen the third dimension plays an important part, but it is felt that the approach presented herein at least opens a new area for thought and analysis and perhaps presages a new era in concrete research. Hereafter in this report the strains referred to as "interior" are those revealed on the sliced plane and should be viewed accordingly.

Results of loading tests conducted at stresses of 2000 psi and 4000 psi are presented in Figs. 8(*a*) and (*b*), respectively. Relative differences between principal strains or "maximum shear strain contours" were determined by analysis of color photographs taken during the tests. The contours of maximum shear strains were traced directly onto a photograph of the specimen as it appeared prior to attachment of the plastic sheet.

Examination of the strain contours at the top surface of the specimen reveals that the major contact area between the steel plate and concrete at 2000 psi (Fig. 8(*a*)) appears to be centered roughly between $\frac{1}{2}$ in. and $2\frac{1}{2}$ in. from the left edge with a minor "high point" centered about $\frac{1}{2}$ in. to $2\frac{1}{2}$ in. from the right edge; at 4000 psi (Fig. 8(*b*)), the corresponding contact areas are between $\frac{1}{4}$ in. and $3\frac{1}{4}$ in. from the left edge and between $\frac{1}{2}$ in. and $1\frac{3}{4}$ in. from the right edge. It should be noted that for both stresses most of the strain contours in the top 5 in. appear to be inclined toward the major contact


Distance from Edges of Specimen, in.
0
1
2
3
2
1
0
Distance from Specimen Extremities, in.
Top
0
2
4
6
8
9
8
6
4
2
0
Bottom
1
2
3
4
5

Maximum Shear Strain*

Number	Microinches per In.
1	50
2	150
3	450
4	800
5	1100
6	1450
7	1830
8	2200
9	2550
10	2900

*Differences between the principal strains.

Maximum Shear Strain*

Number	Microinches per In.
1	50
2	150
3	450
4	800
5	1100
6	1450
7	1830
8	2200
9	2550
10	2900

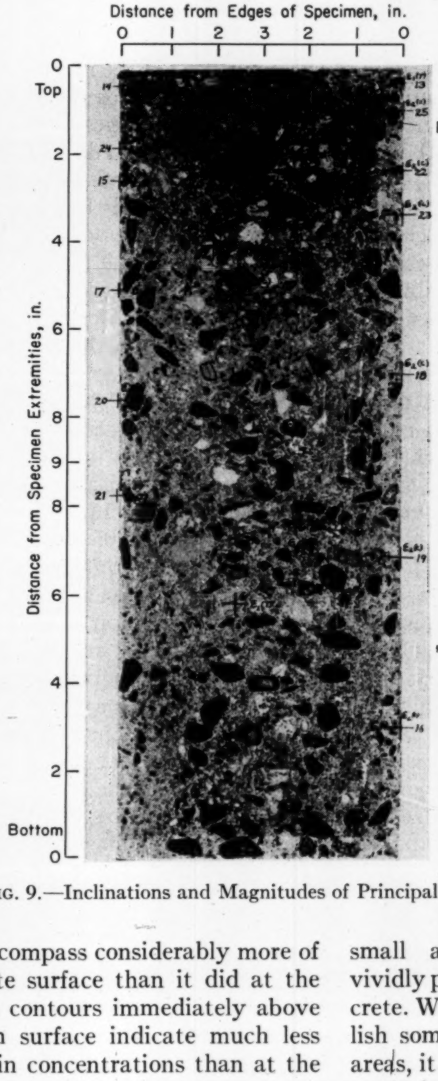
*Differences between the principal strains.

↑ (*a*) Applied stress at 2000 psi. (*b*) Applied stress at 4000 psi.→

FIG. 8.—Maximum Shear Strain Contours from Photoelastic Coating Tests.

area. The top edges on both sides reveal rather low strain levels. Similar strain conditions are observed at the bottom surface of the specimen, although the major steel-to-concrete bearing area appears to encompass considerably more of the concrete surface than it did at the top. Strain contours immediately above the bottom surface indicate much less severe strain concentrations than at the top.

Perhaps the most striking observation to be made after close examination of Fig. 8 is that the strain contours show no partiality between larger and smaller pieces of aggregate. In many instances, the contours actually pass through pieces of large aggregate. The presence of many small areas of strain concentration vividly portrays the heterogeneity of concrete. When an attempt is made to establish some sort of trend regarding these areas, it is found that a trend which may apply for one small sector of the concrete is negated in another sector. Generally speaking, the larger areas of strain con-


Distance from Edges of Specimen, in.
0
1
2
3
2
1
0
Distance from Specimen Extremities, in.
0
Top
2
4
6
8
9
8
6
4
2
Bottom
0
14
24
15
17
20
21
13
25
22
23
18
19
16

Point Number	ϵ_1*	ϵ_2*
1	835T	1495C
2	311C	521C
3	2637C	3083C
4	181T	2839C
5	711C	3726C
6	2087T	257T
7	2785C	4651C
8	989T	1026C
9	582T	688C
10	647T	1158C
11	493T	125T
12	196C	2166C
13	320T	58C
14	356T	64C
15	36T	200C
16	145T	800C
17	157T	868C
18	160T	890C
19	112T	623C
20	145T	810C
21	96T	534C
22	109T	600C
23	6T	36C
24	4T	22C
25	225T	1245C

*ϵ_1 and ϵ_2 are reported in microinches per inch; tensile strains are designated by T and compressive strains by C.

FIG. 9.—Inclinations and Magnitudes of Principal Strains at Selected Points.

centration in Fig. 8(*b*) are at the same locations as those in Fig. 8(*a*), the magnitudes in the former case being greater. Largest areas of high strain concentration are in the vicinity of the ends. The causes of these concentrations are presented in the discussion section of this paper. The high strain contours in the vicinity of the top extend downward between 5 and 6 in. before they extend outward to the full section of the concrete; the higher contours diminish at about 3 in. from the bottom end.

In Fig. 8, presence of higher strain values in the "interior" of the specimens than along the outer boundary reveals an anomaly of concrete strain distribution which may help clarify disagreement between end-to-end strains and strains measured in the central portions along the outer skin. This phenomenon poses the question as to whether strain measured along the outer peripheral surface in the central portion of a concrete specimen actually represents the average strain for the entire specimen, as is usually assumed.

Figure 9 reveals the inclinations and magnitudes of the principal strains at various points selected at random. Quantitative determinations of the principal strains were made with the oblique incidence attachment on all points except 26, 27, and 28. Values of ϵ_1 and ϵ_2 within the concrete mass and along the free vertical boundaries are summarized in the tabulation in Fig. 9 for an applied stress of 4000 psi.

In the vicinity of the top, extensions of the lines indicating inclinations of the longitudinal principal strains reveal that they would pass through the major contact area between the steel and the concrete. Away from the ends, any deviations from expected inclinations of 0 and 90 deg are found in points on pieces of aggregate.

In computing the values for the tabulations in Fig. 9, Poisson's ratio for the concrete was assumed at 0.18. The principal strains, ϵ_1 and ϵ_2, result from and act along the lines of action of σ_1 and σ_2, respectively. For a true uniaxial compressive load, the transverse stress would be zero, but the resistance of the outer shell of the concrete to the natural Poisson movement of the inner core induces a transverse stress, the magnitude and direction of which varies considerably from point to point. In addition, Poisson effects themselves influence both ϵ_1 and ϵ_2. It was expected that the application of compressive stresses would cause, by Poisson effect alone, transverse strains which would be tensile in direction.

Except for a few locations, the induced principal stresses in the transverse direction appear to be fairly low. At points 2, 3, 5, 6, 7, 11, and 12, however, the effects of induced stresses are rather significant. Points 5 and 6 are located on pieces of aggregate and, ironically, indicate opposite phenomena, point 5 having compressive principal strains and point 6 exhibiting tensile principal strains. The stratification of the aggregate particle of point 6 is readily apparent and probably accounts for the anomaly of tensile strains in both directions. Whatever the true explanations may be for the principal strain inclinations at points 5 and 6, it is readily apparent that while they are localized phenomena within the aggregate particles themselves, they do cause strain concentrations in the adjacent areas (Fig. 8).

Points 7 and 12, each located between large pieces of aggregate, reveal the effects of induced transverse stresses which completely mask the expected Poisson effect to the extent that the transverse strains are compressive rather than tensile. This phenomenon in both cases most likely results from interaction of the large aggregate particles between which the two points are situated.

Points 2, 3, and 11 indicate stress-strain manifestations similar to some of those mentioned above, but the inclinations of the principal strains reveal that the left and right segments of the top portion of the specimen were twisted upward and inward toward the specimen axis. As mentioned previously, all points in the upper end indicate the same phenomena, that is, that the top edges have been twisted upward and inward.

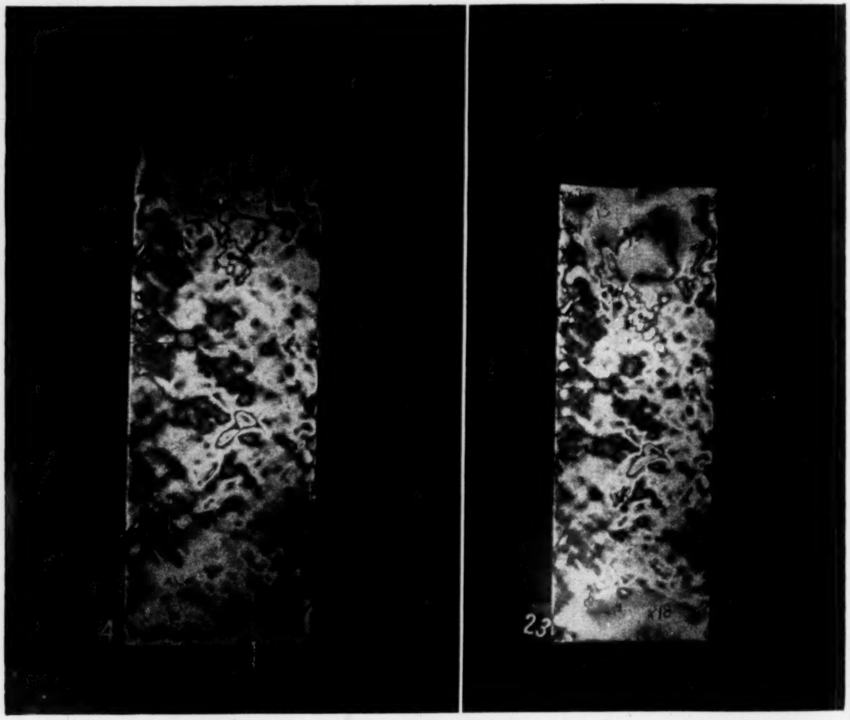
Points 13 and 14 on the vertical edge (free boundary) near the top indicate tensile strains along the vertical boundary. It was noted in earlier sections of this paper that tensile strains were also observed in these areas on the other specimen with resistance strain gages. As a double check, resistance gages ($\frac{1}{4}$ in.) were placed close to points 2 and 3 on this specimen, and again tensile strains were observed. Strain values for points 13 and 14 in the tabulation in Fig. 9 are based upon the uniform assumption of Poisson's ratio equal to 0.18; the validity of this assumption in the vicinity of the ends is problematical. Perhaps it should suffice to say that tensile strains along the vertical boundary were observed at points 13 and 14.

Strains observed at points 13, 25, 22, and 23 reveal drastically changing strain conditions in the top 3 in. of the right side. The axial strains vary from 320 microinches per inch of tensile strain close to the top to a maximum of 1245 microinches per inch of compressive strain and then decrease to 22 microinches per inch compressive strain. The abrupt increase in compressive strain along the free boundary about 1 in. from the top verifies the strain distribution obtained earlier (see Fig. 3). The strain distribution is similar but not as pronounced along the left boundary near the top.

The usual approach to stress analysis of a structural material is to assume that the material is homogeneous with an accompanying constant average value for modulus of elasticity, E. The uncertainty of this approach in the analysis of stress-strain relationships within a concrete mass is apparent from a study of Figs. 8 and 9. Point-to-point strain variations presented in Figs. 8 and 9 would mean, if a constant E is assumed, that the stresses within the mass vary considerably. At points 1 and 12 (Fig. 9) for example, which are located away from the ends and fairly close together, the stress would be 45 per cent higher at point 12 than at point 1. Inasmuch as concrete forms a rather rigid cellular structure, this extreme variation in internal stress is not likely to exist; it seems more logical to state that the strain response is different from point to point within the concrete mass and that E also varies.

As indicated in Figs. 8 and 9, values of ϵ_2 (axial compressive strain) in the vicinity of the top of the specimen are rather high. Presence of high compressive strains in the interior of the specimen near the top surface verifies the statement made earlier that most of the strain computed as being present in the top and bottom 0.10-in. extremities along the outer surface, in actuality, lies in the interior of the concrete beneath the bearing plate. It should be recalled that the end-to-end deformations from which the strains in 0.10-in. segments were computed were obtained with cantilever reeds anchored to rigid top and bottom bearing plates.

The disclosure of high strain concentrations in the extremities leads to the hypothesis that the irregularity of strain distribution throughout the concrete manifests the steel-to-concrete bearing effects. To investigate this premise $\frac{3}{16}$-in. cardboard cushions were placed between the steel bearing plates and the concrete at both ends, and the specimen was loaded as before.[5] Figure 10(*a*) presents maximum shear strain contours ($\epsilon_1 - \epsilon_2$) for a stress of 4000 psi with no cardboard cushion between the steel and concrete (compare with Fig. 8(*b*)); Fig. 10(*b*) pictures strain contours for the same stress with the cardboard cushions in place.



(*a*) Stress of 4000 psi without cardboard. (*b*) Stress of 4000 psi with cardboard.
FIG. 10.—Effects Upon Strain Distribution of Loading Through Cardboard.

Differences in strain distribution between Figs. 10(*a*) and (*b*) are limited largely to the extremities. Strain is much more uniformly distributed along the contact surfaces with the cardboard cushions (Fig. 10(*b*)). The areas of low shear strain visible as black patches near the corners of the extremities in Fig. 10(*a*) are nonexistent in Fig. 10(*b*), revealing that the steel-to-concrete bearing conditions have been altered significantly. Except for the top 3½ in. and the bottom 2 in., the strain contours along the vertical boundaries and in the "interior" of the concrete are virtually identical in Fig. 10(*a*) and 10(*b*). Within reasonable limitations, the condition of the ends of the concrete, with respect to a steel bearing plate, appears to influence very little the strain distribution over most of the specimen.

GENERAL DISCUSSION OF TEST RESULTS

Presence of tensile strains in the specimen extremities reveal interesting possibilities regarding the end conditions of the cylinders. Simplified steel-to-concrete bearing relationships are presented in Fig. 11. With compressive loading and with no friction between steel and concrete, the steel and concrete would each deform axially and transversely independent of the other as indicated in Fig. 11(*b*); the concrete width would increase ($W + \Delta W$) and its length would decrease ($L - \Delta L$), and the steel would behave similarly. Because of the difference between Poisson's ratios of the steel and concrete, 0.3 and 0.18, respectively, the transverse strain in the concrete would be about five times greater than that of

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[5] W. M. Murray and A. J. Durelli, Discussions of "The Effect of Capping Methods and End Conditions before Capping upon the Compressive Strength of Concrete Test Cylinders," by G. E. Troxell, *Proceedings*, Am. Soc. Testing Mats., Vol. 41, pp. 1047–1052 (1941).

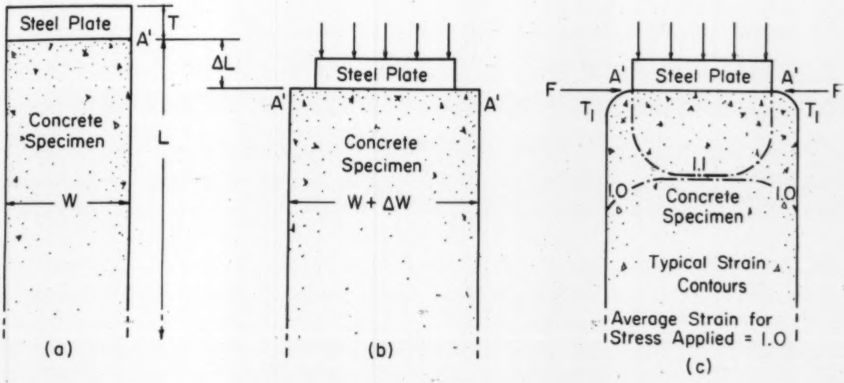
Steel Plate
A'
T
ΔL
Concrete Specimen
L
W
(a)
Steel Plate
A'
A'
Concrete Specimen
W + ΔW
(b)
F
A'
Steel Plate
A'
F
T1
T1
1.1
1.0
1.0
Concrete Specimen
Typical Strain Contours
Average Strain for Stress Applied = 1.0
(c)

FIG. 11.—Friction Effects When Contact Surfaces Are Flat.

(*a*) No applied load and flat contact surfaces.
(*b*) Loaded flat contact surfaces and no friction between surfaces (ideal).
(*c*) Loaded flat contact surfaces with friction between surfaces (actual).

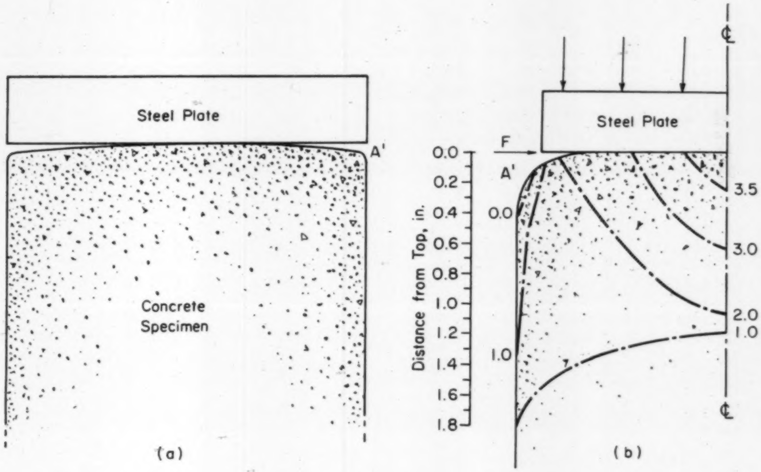
Steel Plate
A'
Concrete Specimen
(a)
℄
Steel Plate
F
A'
0.0
0.2
0.4
0.6
0.8
1.0
1.2
1.4
1.6
1.8
Distance from Top, in.
0.0
1.0
3.5
3.0
2.0
1.0
℄
(b)

FIG. 12.—Combined Effects of Friction and Convexity of the Concrete.

(*a*) Seating load applied with steel surface flat and contact surface convex.
(*b*) Full load applied. Average strain for stress applied = 1.0.

the steel. Under actual conditions, presented in Fig. 11(*c*), these differences in transverse strains result in friction, *F*, at the contact surface of steel and concrete and induce a lateral compressive force.[5] Even with flat contact surfaces between steel and concrete this lateral compression could cause tensile strains, T_1, in the outer skin close to the contact area. Point A^1 is actually "pulled" inward from its original position.

Results in this study revealing tensile strains down as far as 0.5 in. from the top of the specimen seem to indicate that the ends of the specimens were not flat. Figure 12 presents, in exaggerated form, a steel-to-concrete bearing relationship in which the steel surface is flat and the concrete surface is convex. In this situation the lateral compressive force, *F*, would be magnified because the contact stress is greater than the "applied" stress since the steel plate is not in contact with the full concrete cross-section. The bearing contact area between steel and concrete increases as the stress increases until, at some stress, the concrete surface is in full contact with the steel surface. Figure 12 (*b*) pictures the condition for an intermediate stress. The convexity of the concrete magnifies the friction effects and causes extremely high strains in the center of the top surface. If the ends of the concrete were truly convex, the compressive strain concentrations in the vicinity of the contact areas should decrease as the applied stress increases, because the steel plate bears upon more concrete as the concrete deforms, thus decreasing the contact pressure. Examination of the resistance gage data presented in Table VII clearly indicates this phenomenon, particularly in segment *AB*. In segment *LM* the compressive strain did not actually decrease with increasing stress, but the decrease in rate of strain with stress is quite evident. It should be emphasized, however, that the most spectacular differences between mechanical and resistance measurements occur in the top and bottom 1-in. segments. Clarification of the reason for this phenomenon is suggested in Fig. 8 in which the relative strain distribution in the vicinity of the insert (1 in. from top) is vastly different from that nearer the top due primarily to the convexity of the top surface of the concrete. Referring to Table VII, for segment *AB* the relationship between resistance and mechanical values reverses itself as the stress increases; that is, as the steel plate bears upon more and more of the concrete surface, the tendency for strains to concentrate in any one area near the top is diminished. The fact that at 4000 psi the mechanical value is 20 per cent greater than the resistance value suggests undue influence upon the embedded inserts.

Final evidence of convexity of the ends of the concrete specimens used in this study is revealed in the photoelastic coating tests (Figs. 8 and 9). The strain contours near the top are inclined toward a steel-to-concrete bearing area centered roughly between ½ in. and 2½ in. from the left edge of the top for 2000 psi and between ¼ in. and 4 in. for 4000 psi. The greater contact area at 4000 psi, coupled with tensile strains along the vertical boundaries near the top, indicates convexity of the concrete top and bottom surfaces. The convexity, of course, is not symmetrical about the longitudinal axis of the specimen nor is it perfectly regular in curvature.

Convexity of the ends occurred because the early shrinkage rate was extremely high in the low humidity atmosphere (20 per cent RH) in which the concrete was stored;[2] the portion of the concrete first affected is the outer skin. The differential shrinkage strains between inner core and outer skin tend to "draw down" the outer skin at the top and bottom, resulting in convexity of both ends. The degree of convexity of any given specimen depends upon its mix design, its curing history, the length of time exposed to a drying environment, and the relative humidity of the drying atmosphere. In other words, the convexity is a direct function of the shrinkage rate. The ultimate shrinkage in the finished end of the specimen is higher than in the other portions of the specimen due to the introduction of water drawn from below during the finishing process. This implies that the finished end has a higher degree of convexity than the unfinished end, which is believed to be true of the specimens used in this study as revealed by the lower tensile strains in the unfinished end and by the tests utilizing the photoelastic coating technique.

SUMMARY

General belief that strain distribution in concrete is non-uniform has been vividly and conclusively substantiated in this study by the use of resistance strain gages in overlapping gage lengths and by the use of the photoelastic coating technique. Measurements with the shorter resistance gages reveal the localized influences along the outer skin of the dispersion of pieces of larger aggregate within the mortar.

Use of cardboard cushions between the steel and concrete made possible removal of the perceptible effects of convexity of the concrete surfaces. At the same time, frictional resistance between steel and concrete may have been reduced somewhat by the "lubrication effect" of the cardboard. Over most of the concrete, strain distribution was identical regardless of steel-to-concrete relationships in the extremities.

CONCLUSIONS

Because both surface and "interior" concrete strains vary greatly, an end-to-end measurement provides a practical and realistic value for the average strain in an entire specimen.

For central portion measurements, 6-in. resistance gages are more accurate than mechanical gages because they are relatively insensitive to all test variables with the exception of bulging of the specimen.

When mechanical strain gages are used, metal disks (outserts) cemented to the concrete surface serve as better reference points than inserts because they are not affected by internal concrete strains.

Extreme strain fluctuations revealed in this study preclude the use of gages, either mechanical or resistance, having a gage length shorter than 5 in. if average strain of the specimen is desired.

Acknowledgment:

In a research task of this magnitude, the accumulation of reliable and reproducible test data reflects the skill, integrity, and intuitiveness of the project technicians. Resistance strain gage and photoelastic coating installations were made by J. H. McCarthy; data reduction and plotting were accomplished by V. Hernandez. The author gratefully acknowledges the helpful suggestions and counsel of W. A. Shaw and of D. F. Griffin, Director of the Materials Division. Recognition is acknowledged of the significant contribution of the Photographic Division to the successful utilization of the photoelastic coating technique.

DISCUSSION

Mr. H. H. Newlon.[1]—Because the plane face of the semicircular cylinder represents a discontinuity, would you not expect effects from edge stresses which might cause a stress condition different from that existing in the central portion of a true cylinder?

Mr. P. H. Petersen.[2]—Purportedly the top ends of the cylinders in this study were not capped. It is understood that they were wood-floated or steel-trowelled. Therefore, the lack of perfect contact with the top loading plate would likely add to the distortion in the upper $\frac{1}{16}$ to 1-in. length. It would be interesting to know what would have occurred had the top surfaces been capped with an approved capping compound or finished with a cement-rich mortar flattened out by use of a plate-glass cover while the concrete was still semi-plastic. Concrete is seldom loaded to high stresses, especially in prestressed work using surfaces that are wood-floated or even steel-trowelled.

Aside from the above comments, the study is certainly unique. It gives us information on the variation of local stresses in concrete because of nonhomogeneity due to the presence of gravel, sand, and paste. From a structural design standpoint, however, there is nothing in this paper which indicates that we should eliminate our one hypothesis that concrete should be considered as a homogeneous material.

Mr. J. R. Keeton (*author's closure by letter*).—As stressed in the text, maximum shear strain measurements made on the face of the sliced specimen do not reflect true three-dimensional strains. The principal value of such a test is the observation of the relative effects upon strain distribution of the coarse aggregate, fine aggregate, and mortar.

It is correct that no capping material was used on any of the specimens tested in this study The photoelastic coating test in which cardboard was placed between loading plates and concrete, however, revealed that uniformity of load application across the ends affected the strain distribution only within about 3 in. of the ends. Inasmuch as this study dealt principally with concrete strain distribution and with methods of measuring concrete strains, it should be considered as a research effort intended to stimulate development of new approaches and ideas in the science of engineering materials.

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[1] Virginia Council of Highways, Investigation and Research, Charlottesville, Va.

[2] The Master Builders Co., Cleveland, Ohio.

A METHOD FOR DETERMINING MECHANICAL RESONANCE FREQUENCIES AND FOR CALCULATING ELASTIC MODULI FROM THESE FREQUENCIES*

BY S. SPINNER[1] AND W. E. TEFFT[1]

SYNOPSIS

Techniques for exciting. detecting, and measuring the mechanical resonance frequencies of solid specimens are described in Part I. In addition, relations are given in Part II for computing the appropriate elastic moduli from these resonance frequencies.

The method of obtaining the elastic moduli of solid bodies from their mechanical resonance frequencies, originated about 25 years ago (1,2),[2] has proved to be a very fruitful one. This is attested by the numerous investigators who have successfully utilized this method since its inception. Through the intervening years these investigators have improved and refined the basic method so that today resonance frequencies can be determined to a high degree of accuracy. This refinement in experimental techniques has been accompanied by a corresponding refinement in the theoretical relations for calculating the various elastic moduli from the appropriate resonance frequencies.

This paper has a twofold purpose. The first is to describe the techniques for exciting, detecting, and measuring the mechanical resonance frequencies of specimens. These techniques are dealt with in Part I. The second purpose, dealt with in Part II, is to describe the methods for calculating the various elastic moduli from the appropriate resonance frequencies. The techniques described in Part I are applicable to any material that is sufficiently elastic[3] to be vibrated in resonance. If the material is also homogeneous, isotropic,[4] and of the appropriate shape, then the computations described in Part II are applicable. If the conditions of isotropy, homogeneity, and proper shape are not fulfilled, then the various resonance frequencies may still be determined even though the equations in Part II would not apply. These resonance frequencies have many useful diagnostic applications (for quality control, for instance) even when the elastic moduli are not calculated from them.

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* Presented at the Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] Physicist, Engineering Ceramics Section, National Bureau of Standards, Washington, D. C.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

[3] By the term "elastic" is meant that if the body is stressed within the elastic limit, it will deform instantaneously and uniformly, in accordance with the stress, and once the stress is released the body will return instantaneously to its original size and shape (without loss of energy). It is recognized that probably no material conforms ideally to this condition. However, many solids conform sufficiently closely so that the figures of accuracy given in the text are applicable.

[4] The term "isotropic" as used here means that the elastic properties are the same in all directions in the material. Under such a condition two elastic constants are sufficient to completely describe the material elastically. Strain-free glass conforms most closely to this condition in practice. If the material is completely composed of polycrystalline grains or, like some whitewares, of polycrystalline grains in a glassy matrix and the grains are randomly distributed and oriented, then the condition of isotropy will be fulfilled macroscopically even though the individual grains are anisotropic, and the equations based on this condition still apply to the bulk material.

The method to be described is often referred to as "sonic" because the resonance frequencies involved are usually in the sonic range (up to about 20 kc). However, it is believed that the term "resonance method" is more accurate because it is more descriptive of the primary characteristic of the method and also because it would still be appropriate, even if the frequency were extended beyond the sonic range. This latter situation has, indeed, already developed and will undoubtedly continue as equipment and experimental techniques improve.

It is intended to describe the proposed method in such a manner that although the sonic range of frequencies is usually involved, higher frequencies are not excluded.

PART I: METHODS FOR DETERMINING MECHANICAL RESONANCE FREQUENCIES OF SOLIDS[5]

SPECIMENS

The equations in Part II are developed for specimens in the shape of cylindrical rods or rectangular bars. Therefore, if the elastic moduli are to be calculated from the resonance frequencies, the specimens should be of one of these two shapes.

Size and shape of the specimens usually depend on the material being tested. The dimensions of the specimens should be large enough so that their different fundamental resonance frequencies will be within the range of the equipment (mainly the transducers) but not so large as to be too cumbersome to fabricate or handle experimentally.

For specimens consisting of composite materials like concrete, the smallest dimension should be large (by a factor of at least 3) compared to the largest pieces of aggregate to satisfy the criteria of macroscopic homogeneity and isotropy.

A convenient size for such materials as glass, ceramics, or steel has been found to be about 6 by 1 by $\frac{1}{2}$ in. for rectangular bars, about 6 by $\frac{1}{2}$ by $\frac{1}{2}$ in. for square bars, and about 6 in. long and $\frac{1}{2}$ in. in diameter for cylinders, but considerable latitude in the values of these dimensions is permissible. Widely used sizes for laboratory specimens of concrete are 16 by 3 by 4 in. and 16 by $3\frac{1}{2}$ by $4\frac{1}{2}$ in., but beams as large as 60 by 5 by 5 in. and 48 by 6 by 6 in. have been successfully resonated. Concrete cylinders, which usually have a length-diameter ratio of 2 to 1, are regularly tested by this method. For rectangular specimens the ratio of length to either cross-sectional dimension should probably not be less than 3 to 1.

EQUIPMENT

The entire equipment consists of standard components which are readily available. These components are:

(*a*) Audio oscillator—should have continuous frequency control from about 60 cycles to at least 20 kc. The output should be reasonably flat. Frequency should not vary by more than a fraction of a cycle in about $\frac{1}{2}$ min for any control setting.

(*b*) Audio amplifier—should have a maximum output of at least 20 w. Frequency range and flatness of response should be at least as good as the oscillator.

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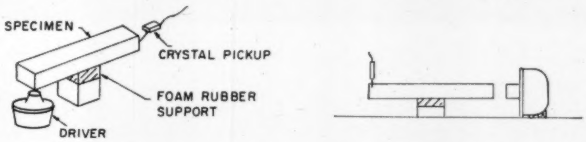
[5] Part I was written by S. Spinner.

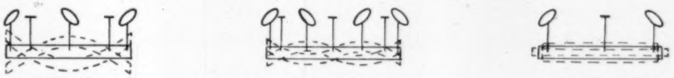
(c) Pickup amplifier—should be able to serve as a pre-scope amplifier and match impedance with whatever type of pickup transducer is used. (Components *a*, *b*, and *c* should have a continuous gain control.)

(*d*) Cathode ray oscilloscope.



POSITION OF DRIVER AND SPECIMEN FOR TORSIONAL VIBRATIONS

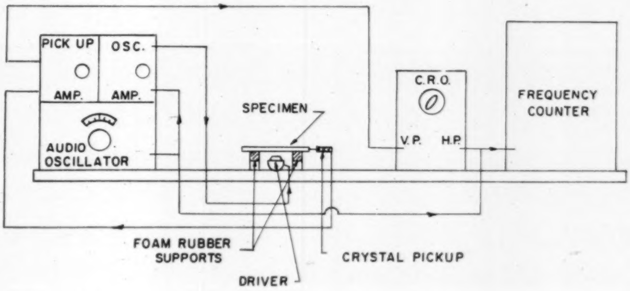
POSITION OF DRIVER AND SPECIMEN FOR LONGITUDINAL VIBRATIONS



FLEXURAL FUNDAMENTAL

FLEXURAL FIRST OVERTONE

LONGITUDINAL FUNDAMENTAL



POSITION OF DRIVER AND SPECIMEN FOR FLEXURAL VIBRATIONS

FIG. 1.—Diagrammatic Sketch of Setup Used for Dynamic Resonance Measurements.

(*e*) Frequency counter—should be able to measure frequencies to one part in last significant figure indicated on the instrument (that is, 1 cps on the 1-sec interval or 0.1 cps on the 10-sec interval). Beckman models No. 7360 or No. 7380 or Hewlett Packard Model No. 5238 or equivalent will serve.

(*f*) Two transducers, one used as a driver and the other as a pickup. The driver may be a speaker (one of the tweeter type is desirable) or a record-cutting head or similar device depending on the method used for coupling the energy of the driver to the specimen. For more massive specimens, especially those having high internal damping, such as concrete, a heavier duty speaker of fairly wide range is needed. The speaker should be able to handle the full power output of the driver amplifier.

The pickup may be a phonograph cartridge of the crystal or magnetic reluctance type.

The frequency response of both transducers is generally the limiting factor of the frequency response of the entire equipment.

METHOD OF OPERATION

As indicated earlier, the purpose of the equipment is to excite and detect resonance in the specimen and measure the frequency of this resonance. The method by which this is accomplished may be understood by referring to the diagrammatic sketch in the lower part of Fig. 1.

The output of the audio oscillator is amplified and then fed to the driver, whose mechanical energy in turn is transmitted to the specimen. (Different methods of transmitting the energy from the driver to the specimen and from the specimen to the pickup are described in the next section.) At the same time that the driver is being energized, the oscillator output also goes to the horizontal plates of the scope. When the oscillator output is not near a resonance frequency of the specimen, then all that is seen on the scope is a horizontal line (which is actually the electron beam moving back and forth at the frequency of the oscillator).

As the oscillator frequency is scanned, however, it eventually reaches one of the mechanical resonance frequencies of the specimen. The predominant characteristic of a system (in this case, the specimen) in resonance is the large increase in the amplitude of its vibrations. This occurs in the specimen at its mechanical resonance frequencies. This increased amplitude is detected by the pickup, in contact with the specimen, also amplified, and then fed to the vertical plates of the scope. If the same frequency is received by the horizontal and vertical plates of the scope at the same time, a Lissajou pattern, as drawn in the diagram, results. The oscillator frequency setting at which this pattern has its maximum vertical amplitude is one of the resonance frequencies of the specimen. The purpose of the frequency counter, also connected to the oscillator, is to determine this frequency more accurately than is possible by reading the oscillator scale itself. The counter also does away with the necessity for calibrating the oscillator. If a frequency counter is not available, the equipment may still be used but the accuracy of the resonance frequency determination will usually be reduced by a factor of about 100.

METHODS OF COUPLING

The methods of coupling the energy from the driver to the specimen and detecting the vibrations of the specimen with the pickup should be such that although sufficient energy is transmitted to the specimen that its resonance vibrations can be easily detected, nevertheless the degree of coupling should not be such that the driver or pickup loads the specimen and affects its natural resonance frequency. In practice, this undesirable loading is a more serious problem with the driver than with the pickup. The evidence for this undesirable loading is that a change in the amplitude of vibrations of the driver results not only in a change in the amplitude of the specimen vibrations at resonance but also in an appreciable change in the frequency of the resonance vibrations of the specimen. Also a shift in the relative position of driver and pickup will usually result in a change in the apparent resonance frequency.

The following methods for coupling the energy from the driver to the specimen and detecting these vibrations have been found to be quite satisfactory for materials having low damping (for example, steel, glass, ceramics) since, the loading of the specimen being reduced to a minimum, they conform to the conditions of the opening paragraph of this section. The evidence for this is that the resonance frequencies of the specimens obtained in this way are highly reproducible, and also, that changes in the amplitude of vibration of the driver cause no noticeable change in the measured resonance frequencies of the specimens.

(*a*) The specimen rests on foam rubber

at nodal points and the vibrations of the speaker are transmitted through air to the specimen. The resulting vibrations of the specimen are detected by the pickup which touches very lightly against the specimen. This is illustrated in the upper portion of Fig. 1. (The different types of resonance frequencies, both fundamental and overtones, can be excited and detected by appropriate placement of the driver and pickup with respect to the specimen. These different types of vibrations will be discussed in detail later.)

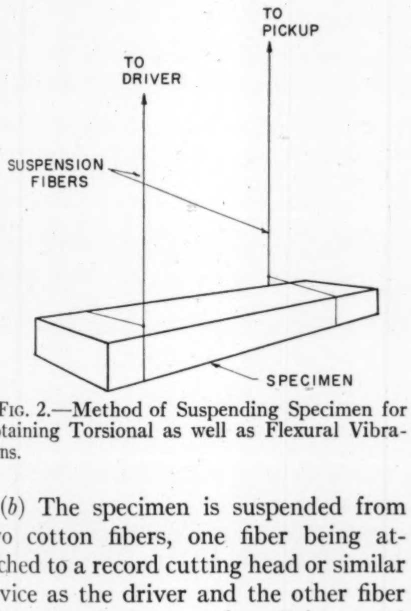


Fig. 2.—Method of Suspending Specimen for Obtaining Torsional as well as Flexural Vibrations.

(*b*) The specimen is suspended from two cotton fibers, one fiber being attached to a record cutting head or similar device as the driver and the other fiber being attached to a (usually) crystal pickup. If the fibers are attached to opposite sides of the specimen as shown in Fig. 2, then not only will the flexural resonance frequencies of the specimens be obtained but also the torsional.

(*c*) This method combines certain features of the first two. The specimen is suspended as in the second method, but it is driven through air with a tweeter and the vibrations detected with a pickup as in the first method.

(*d*) Another variation consists in having the specimen suspended from two fibers, one fiber being attached to the pickup but the specimen being driven through air by means of the tweeter. The resonance vibrations are detected by the pickup attached to the fiber.

Methods (*b*) and (*d*) are generally limited to lighter specimens. However, for lighter and smaller specimens, these two methods, especially method (*b*), will sometimes give reliable results when method (*a*) fails. Methods (*a*) and (*c*) are the most versatile in that the most freedom is allowed in the relative placements of driver and pickup with respect to the specimen to obtain all possible types of vibration.

Suspension methods are most easily adaptable for measurements at elevated temperatures.

(*e*) For materials such as concrete where more energy must be transmitted to the specimens to excite resonance, it has often been found necessary to use the following method: The specimen rests on foam rubber at nodal points or entirely on a thick foam rubber pad, and the vibrations are transmitted directly through a rod cemented to the voice coil of the speaker and bearing on the appropriate part of the specimen. This method applies to massive specimens which will vibrate freely resting on a pad if the latter is sufficiently thick and spongy. The pressure of the driver against the specimen should be adjustable to get the best response. This may be accomplished by mounting the speaker on a counterbalanced arm under the table with the rod extending through a hole in the table. A thumbscrew and spring may be used to depress and raise the mechanism to adjust the pressure. The vibrations are detected by the pickup which is placed against the end of the specimen.

ACCURACY

The over-all damping of the entire system, including the specimen, driver, pickup, and coupling mechanism, is generally so small that the accuracy of the resonance frequency measurements approaches the accuracy of the frequency counter. This is 1 cycle for the 1-sec gate of the counter and 0.1 cycle for the 10-sec gate. Generally the 1-sec gate is sufficient for higher resonance frequencies (say from about 5 kc and above) and the 0.1-sec gate may be used for resonance frequencies below 5 kc. For specimens vibrated by one of the suspension methods, it is found that the position of the suspension with respect to the nodal points may change the resonance frequency by about 2 cycles in 5000. The best results will be obtained when the suspensions are nearest the nodal points. (At the nodal points, of course, the effective response is zero.) In general, the measured resonance frequencies of bulkier specimens are least affected by the method of coupling or the position of the supports with respect to the nodes. In view of all these considerations a conservative generic estimate of the accuracy of the resonance frequencies is about 1 part in 4000. This estimate applies where methods of coupling (*a*) through (*d*) are used. For method (*e*) the accuracy is usually lower than this.

The fact that the resonance frequencies can be determined to this accuracy does not necessarily mean that the elastic moduli calculated from them (as described in Part II) will be of the same order of accuracy. This result can only be achieved if the other parameters involved in the determination of the elastic constants, namely, density and dimensions, are known to an accuracy comparable with the resonance frequencies. In general the limiting factor in the accuracy of the calculated values of elastic moduli is the uniformity and accuracy of the dimensions.

IDENTIFICATION OF DIFFERENT TYPES OF VIBRATION

Specimens of the shapes indicated previously may be caused to resonate in different ways—longitudinally, flexurally (or transversely), and torsionally. The rectangular bars may vibrate in flexural resonance both flatwise and edgewise. In addition to the fundamental resonance frequencies, higher overtones of these different types of vibration are theoretically possible and may be obtained experimentally, providing the frequencies are not above the range of the equipment.

Young's modulus is related to the longitudinal and flexural resonance vibrations and the shear modulus is related to the torsional. Since for the cylinders, two, and for the rectangular bars, three separate determinations of Young's modulus may be made from fundamental modes, a convenient means presents itself for checking the internal consistency of the data, namely, by noting the agreement in Young's modulus as calculated from the different resonance frequencies for a particular specimen. Conversely, failure to obtain agreement in Young's modulus calculated from these separate resonance frequencies would indicate a lack of isotropy or homogeneity in the specimens.

When a specimen vibrates in resonance, the type of vibration and the overtone thereof may be determined unequivocally by "probing." That is, the pickup is held against different parts of the specimen while it is vibrating in resonance and the resulting pattern of changes in the Lissajou figures seen on the scope establishes the type and overtone of the particular resonance frequency. This procedure can be demonstrated most readily by referring once more to method of coupling (*a*) shown in Fig. 1.

Consider first a specimen vibrating in the fundamental flexural resonance frequency. In this case, the ends of the specimen are oscillating in the same direction at the same time (in phase), the center of the specimen is oscillating in the opposite direction at the same time (180 deg out of phase) and, at the nodal points, about one fourth of the length of the specimen in from each end, the motion is at a minimum. Now, if the pickup is held against the side of the specimen as its length is traversed in short steps from one end to the other, then the pattern of phase relations of the Lissajou figures as seen on the scope will vary in a manner exactly corresponding to the mechanical phase relations just described.

Thus, the pattern of Lissajou figures drawn immediately above the different portions of the specimen represent equally well the electrical phase relations that would be seen on the scope if the pickup were held against that portion of the specimen or the mechanical phase relations existing among the same portions of the specimen. At intermediate positions of the specimen between the nodes and antinodes, the pattern on the scope will also take intermediate positions in amplitude and there will be a reversal in phase as the node is crossed.

This illustrates the general rule for determining by probing the manner in which the specimen is vibrating: namely, *the electrical phase relationships of the Lissajou figures seen on the scope as the pickup is held against the different parts of the specimen are an exact reflection of the mechanical phase relationships existing in the specimen while it is in resonance.* It is not important whether the maxima in the Lissajou patterns, occurring when the pickup is held against the ends of the specimen, are diagonal to the left or right. The direction of the diagonal is determined by the particular characteristics of the scope and the angle of the pickup as held against the specimens. In fact, if the Lissajou pattern is perfectly vertical, it may be made diagonal by slightly detuning the oscillator or by changing the angle of the pickup against the specimen. It is most important, however, that once a certain diagonal of the Lissajou pattern is established, that the pickup be moved parallel to itself as it traverses the specimen so that the changes in Lissajou patterns will truly reflect the mechanical changes occurring in the specimen and not be confused by additional changes introduced by altering the angle of the pickup as held against the specimen.

Furthermore, if none of the settings of the equipment is altered during the course of probing, then the vertical amplitude of the responses seen on the scope will also be in proportion to the amplitude of the mechanical vibrations of the portion of the specimen against which the probe is held. Thus, for flexural vibrations, the amplitude of the Lissajou figures seen on the scope when the pickup is held against the center of the specimen will be somewhat lower than when the pickup is held against the ends of the specimen. This is in conformity with the actual mechanical vibrations of the same portions of the specimens, since it is known that the maximum displacement from the neutral position of the ends of a bar vibrating in flexural resonance is greater than that at the center. However, the maximum amplitude of these relationships is of secondary importance in comparison with the phase relationships in determining the type and overtones of the resonance vibrations of the specimen.

A specimen vibrating at the first overtone of flexural resonance will have the opposite ends moving out of phase, there will be three nodal points, and the other two maxima will be out of phase with the ends as illustrated in the figure. These will also correspond to the Lissajou patterns shown immediately above the specimen in Fig. 1 when the pickup is held against the indicated portion of the specimen.

A specimen vibrating in fundamental longitudinal resonance will have the opposite ends out of phase mechanically (moving in opposite directions at the same time) and a nodal point at the center. Here also, the Lissajou patterns shown above the specimen will correspond exactly to the mechanical phase relationships existing in the specimen.

The most favorable position of the driver and pickup with respect to the specimen for obtaining torsional vibrations is also shown, although the manner in which the specimen actually vibrates is not illustrated (being somewhat simpler to describe than to draw).

In the fundamental of torsional resonance the opposite ends twist in opposite directions at the same time (that is, while one end twists clockwise the other end twists counterclockwise, and vice versa) and there is a node at the center of the specimen.

Therefore, if only the side of the specimen were traversed by the pickup, the sequence in the pattern of Lissajou phase changes could not easily be distinguished from that occurring when the specimen resonates in the fundamental, longitudinal frequency. The torsional may be distinguished from the longitudinal mode of vibration, however, if it is recalled that in torsion the opposite cross-sectional faces twist, whereas for the longitudinal mode these opposite faces move toward and away from the center without twisting. Therefore, if the end of the specimen is traversed by the pickup there will be a phase reversal from one corner to the diagonal one of this (sectional) face when the specimen is in torsional resonance, while no phase change will occur in the corresponding traversal of the pickup when the specimen resonates longitudinally.

Similarly the first overtone of torsional resonance cannot easily be distinguished from the fundamental of flexure as long as only the long side of the specimen is traversed by the pickup, as the mechanical (and consequently electrical) pattern of phase changes will be alike. However, the two can be distinguished by traversal of the opposite cross-sectional faces as here, too, the torsional mode will show an electrical phase reversal corresponding to the mechanical one and the flexural will show no such phase reversal.

The first overtone of the longitudinal mode can be distinguished from the fundamental of flexure by noting the directions in which the vibrating parts of the specimen are moving. This can easily be ascertained by observing the position in which the pickup must be held in relation to the specimen in order to obtain a maximum indication on the scope. Most phonograph pickups are presently designed to give a maximum response when the vibratory movement (causing the response) is at right angles to the vertical plane through the long axis of the cartridge. Thus, in Fig. 1 the pickup is shown in the best position to give a maximum response for the particular mode of vibration indicated. For the longitudinal modes, the long axis of the cartridge is seen to be held at right angles to the long axis of the specimen, while for the flexural mode the long axis of the cartridge is in the same plane as the long axis of the specimen. In addition, the first overtone of the longitudinal mode occurs at a much higher frequency than the fundamental flexural one.

In general, if one keeps in mind the manner in which the specimen is vibrating as a whole, then the corresponding electrical pattern seen on the scope will reveal some distinguishing characteristic by which the different types of vibration can be uniquely identified. Once the type of vibration has been established by probing and the nominal value of the resonance frequency of that particular type determined, then the resonance frequency of many specimens of the same type and dimensions vibrating in the same mode may be determined more rapidly without going through the probing procedure in each case.

Also, if this concept of the correspondence of the electrical and mechanical phase relationships is kept in mind, then it can be applied, although in a more limited way, when resonance is obtained by a suspension method such as method of coupling (*b*). For instance, a torsional can be distinguished from a flexural mode by noting whether the response on the scope continues or dies out as the points of suspension are moved from the sides toward the center, that is, toward the long axis of the specimen. Thus, the response of a specimen suspended as shown in Fig. 2 will continue for the flexural mode but will be noticeably reduced for a torsional mode if this procedure is followed. Similarly, nodal points can be located by moving the points of suspension along the long axis of the specimen.

For illustration, only the rectangular bars were used. However, the same principles apply to cylindrical specimens or, for that manner, to any other shape resonating in any particular mode. The method just described may be regarded as a more exact extension of the principle of Chladini figures.

Acknowledgment:

The author is indebted to R. C. Valore, Jr., formerly of the National Bureau of Standards staff, for his contributions in developing many of the refinements, especially the technique of probing, described in this paper.

PART II: EQUATIONS FOR COMPUTING ELASTIC MODULI FROM MECHANICAL RESONANCE FREQUENCIES[6]

For cylinders, highly accurate theoretical equations have been worked out for each of the three basic types of resonance vibrations; these are longitudinal, flexural (or transverse), and torsional resonance. For prismatic bars, however, the existing theories are only approximate. If a high degree of accuracy is desired, it is therefore necessary to use cylindrical specimens. If, on the other hand, errors of the order of 1 per cent can be tolerated, the experimental difficulties will be reduced by the use of prismatic bars.

If Poisson's ratio is unknown, Young's modulus may be approximately determined from the density, dimensions, and resonance frequency of longitudinal or flexural vibrations. For either of these types of vibration, however, the equations for computing Young's modulus contain correction factors which vary somewhat with the value of Poisson's ratio. It is thus necessary in high precision work to know the value of Poisson's ratio. This quantity may be determined as follows:

If the torsional resonance frequency is measured, the shear modulus may then be computed from the equations given later in the section on torsional resonance. An approximate value of Young's modulus is then determined from the flexural or longitudinal resonance frequency and the use of the equations of the sections on flexural resonance or longitudinal resonance. Then from the well-known equation

$$\mu = \frac{E}{2G} - 1 \ldots\ldots\ldots\ldots (1)$$

where:

μ = Poisson's ratio,
E = Young's modulus, and
G = shear modulus

an approximate value of μ is obtained. If this value of μ is then used to redetermine the correction factors, a better value of E and then μ is obtained. Repetition of this procedure converges rapidly to the correct values of E and μ.

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[6] Part II was written by W. E. Tefft.

In the discussion which follows, the following notation will be used:

ρ = density of the material,
l = length of the specimen,
d = diameter of cylindrical specimens,
a and b = cross-sectional dimensions of prismatic specimens, with the restriction $a \leq b$,
t = cross-sectional dimension in the direction (or plane) of vibration (flexure of prisms only),
n = 1, 2, 3, $\cdots$ = order of vibration, and
f_n = resonance frequency of the nth mode of vibration. If the symbol f appears without a subscript, the fundamental mode ($n = 1$) is assumed.

Since the numerical factors in all equations which follow are dimensionless, any consistent system of units may be used. Thus, for example, if the cgs system is used, densities in grams per cubic centimeter, lengths in centimeters, and frequencies in cycles per second give the moduli in dynes per square centimeter. In the English system, one might use the units: density in pounds per cubic inch, lengths in inches, and frequencies in cycles per second. This would give the moduli in $\frac{\text{lb}}{\text{in. sec}^2}$. To convert this to pounds per square inch divide the result by the acceleration of gravity, 386.09 in. per sec^2.

It is emphasized that the accuracies to be given subsequently in describing the equations are those inherent in the equations themselves, and not those of the experimental results to which these equations apply. In fact, the general goal is to make the equations more accurate than the best reasonably achievable experimental data. Therefore, the limitation on the accuracy of the experimental results will usually be the measured resonance frequencies, densities, and especially the dimensions of the specimens, as explained in Part I in the section on identification of different types of vibrations.

FLEXURAL RESONANCE

Cylinders:

The basic equations for computing Young's modulus from the first three modes of flexural resonance of cylinders (3) are:

$$E = 1.261886 \frac{\rho l^4}{d^2} f_1^2 T_1 \quad \text{.........(2)}$$

$$E = 0.1660703 \frac{\rho l^4}{d^2} f_2^2 T_2 \quad \text{.........(3)}$$

$$\text{and } E = 0.04321184 \frac{\rho l^4}{d^2} f_3^2 T_3 \quad \text{........(4)}$$

where T_1, T_2, and T_3 are the correction factors given in Tables I, II, and III respectively. These correction factors are functions of d/l and μ only.

The following method of interpolation in these tables is strongly recommended. Compute the quantity $(d/l)^2$ for the specimen being measured, and for the two entries in the table having d/l values nearest to that of the specimen. Label these quantities x, x_2 and x_1 respectively. Choose the two nearest values of μ above and below the assumed μ of the specimen, labeling them μ_2 and μ_1 respectively. Compute the correction factors for μ_1 and μ_2 at the d/l of the specimen from the equations

$$T_n(x, \mu_1) = \frac{T_n(x_2, \mu_1) - T_n(x_1, \mu_1)}{x_2 - x_1} \cdot (x - x_1) + T_n(x_1, \mu_1) \quad \text{....(5)}$$

$$\text{and } T_n(x, \mu_2) = \frac{T_n(x_2, \mu_2) - T_n(x_1, \mu_2)}{(x_2 - x_1)} \cdot (x - x_1) + T_n(x_1, \mu_2) \quad \text{....(6)}$$

TABLE I.—CORRECTION FACTOR FOR THE FUNDAMENTAL MODE OF FLEXURAL VIBRATION OF CYLINDERS, T_1, AS A FUNCTION OF THE DIAMETER TO LENGTH RATIO, d/l, AND POISSON'S RATIO, μ.

Diameter to Length Ratio, d/l	Poisson's Ratio, μ										
	0.00	0.05	0.10	0.15	0.20	0.25	0.30	0.35	0.40	0.45	0.50
0.00	1.000000	1.000000	1.000000	1.000000	1.000000	1.000000	1.000000	1.000000	1.000000	1.000000	1.000000
0.02	1.001954	1.001979	1.002004	1.002029	1.002053	1.002077	1.002100	1.002124	1.002147	1.002170	1.002193
0.04	1.007804	1.007905	1.008004	1.008102	1.008199	1.008295	1.008388	1.008482	1.008575	1.008666	1.008757
0.06	1.017522	1.017748	1.017968	1.018186	1.018405	1.018619	1.018826	1.019038	1.019245	1.019450	1.019653
0.08	1.031064	1.031461	1.031848	1.032233	1.032618	1.032994	1.033360	1.033733	1.034096	1.034459	1.034818
0.10	1.048367	1.048983	1.049580	1.050174	1.050765	1.051344	1.051916	1.052484	1.053050	1.053610	1.054170
0.12	1.069357	1.070225	1.071077	1.071920	1.072753	1.073577	1.074393	1.075202	1.076008	1.076808	1.077604
0.14	1.093950	1.095111	1.096256	1.097378	1.098495	1.099599	1.100694	1.101782	1.102864	1.103941	1.105015
0.16	1.122061	1.123544	1.125007	1.126452	1.127884	1.129302	1.130711	1.132113	1.133509	1.134901	1.136288
0.18	1.153592	1.155430	1.157245	1.159039	1.160817	1.162582	1.164337	1.166086	1.167830	1.169569	1.171308
0.20	1.188458	1.190676	1.192869	1.195038	1.197191	1.199332	1.201464	1.203591	1.205714	1.207838	1.209961
0.22	1.226567	1.229189	1.231781	1.234351	1.236906	1.239449	1.241986	1.244521	1.247057	1.249596	1.252141
0.24	1.267838	1.270881	1.273894	1.276886	1.279865	1.282836	1.285807	1.288779	1.291757	1.294746	1.297747
0.26	1.312189	1.315668	1.319120	1.322555	1.325980	1.329403	1.332832	1.336270	1.339723	1.343194	1.346687
0.28	1.359546	1.363477	1.367381	1.371276	1.375167	1.379065	1.382977	1.386911	1.390869	1.394857	1.398879
0.30	1.409842	1.414233	1.418606	1.422974	1.427352	1.431747	1.436169	1.440625	1.445121	1.449661	1.454250
0.32	1.463014	1.467873	1.472724	1.477584	1.482465	1.487380	1.492337	1.497345	1.502411	1.507540	1.512737
0.34	1.519006	1.524340	1.529680	1.535043	1.540446	1.545901	1.551420	1.557012	1.562682	1.568439	1.574286
0.36	1.577766	1.583580	1.589417	1.595298	1.601240	1.607259	1.613367	1.619572	1.625884	1.632309	1.638853
0.38	1.639253	1.645548	1.651888	1.658300	1.664800	1.671405	1.678129	1.684983	1.691975	1.699113	1.706401
0.40	1.703422	1.710202	1.717054	1.724007	1.731082	1.738298	1.745669	1.753207	1.760921	1.768818	1.776904
0.42	1.770243	1.777505	1.784874	1.792382	1.800052	1.807904	1.815954	1.824214	1.832694	1.841401	1.850342
0.44	1.839681	1.847424	1.855317	1.863393	1.871677	1.880193	1.888956	1.897979	1.907273	1.916846	1.926702
0.46	1.911712	1.919934	1.928355	1.937012	1.945932	1.955140	1.964653	1.974485	1.984646	1.995143	2.005980
0.48	1.986311	1.995009	2.003964	2.013216	2.022795	2.032727	2.043030	2.053718	2.064802	2.076288	2.088178
0.50	2.063458	2.072628	2.082123	2.091985	2.102247	2.112937	2.124075	2.135673	2.147741	2.160285	2.173305
0.52	2.143136	2.152775	2.162814	2.173303	2.184276	2.195762	2.207781	2.220345	2.233465	2.247142	2.261377
0.54	2.225331	2.235431	2.246024	2.257157	2.268871	2.281194	2.294146	2.307741	2.321984	2.336876	2.352415
0.56	2.310030	2.320588	2.331739	2.343539	2.356026	2.369231	2.383174	2.397866	2.413311	2.429508	2.446448
0.58	2.397222	2.408232	2.419951	2.432439	2.445736	2.459873	2.474869	2.490733	2.507467	2.525064	2.543511
0.60	2.486902	2.498357	2.510654	2.523855	2.538002	2.553126	2.569244	2.586362	2.604478	2.623578	2.643647

TABLE II.—CORRECTION FACTOR FOR THE FIRST OVERTONE OF FLEXURAL VIBRATION OF CYLINDERS, T_2, AS A FUNCTION OF THE DIAMETER TO LENGTH RATIO, d/l, AND POISSON'S RATIO, μ.

Diameter to Length Ratio, d/l	Poisson's Ratio, μ										
	0.00	0.05	0.10	0.15	0.20	0.25	0.30	0.35	0.40	0.45	0.50
0.00	1.000000	1.000000	1.000000	1.000000	1.000000	1.000000	1.000000	1.000000	1.000000	1.000000	1.000000
0.01	1.001352	1.001376	1.001399	1.001422	1.001445	1.001467	1.001489	1.001511	1.001533	1.001554	1.001576
0.02	1.005404	1.005499	1.005592	1.005683	1.005774	1.005863	1.005952	1.006039	1.006126	1.006212	1.006297
0.03	1.012144	1.012355	1.012564	1.012770	1.012973	1.013174	1.013373	1.013569	1.013763	1.013957	1.014149
0.04	1.021552	1.021926	1.022295	1.022660	1.023019	1.023376	1.023728	1.024077	1.024421	1.024763	1.025103
0.05	1.033605	1.034188	1.034760	1.035326	1.035885	1.036440	1.036987	1.037530	1.038064	1.038597	1.039128
0.06	1.048267	1.049105	1.049924	1.050734	1.051537	1.052327	1.053108	1.053883	1.054653	1.055417	1.056178
0.07	1.065511	1.066641	1.067750	1.068844	1.069926	1.070995	1.072054	1.073103	1.074146	1.075181	1.076211
0.08	1.085296	1.086759	1.088198	1.089613	1.091015	1.092401	1.093775	1.095137	1.096491	1.097837	1.099177
0.09	1.107583	1.109415	1.111219	1.112996	1.114756	1.116497	1.118223	1.119937	1.121641	1.123337	1.125025
0.10	1.132330	1.134568	1.136772	1.138949	1.141103	1.143235	1.145352	1.147455	1.149547	1.151630	1.153705
0.11	1.159496	1.162177	1.164820	1.167429	1.170012	1.172573	1.175115	1.177643	1.180161	1.182668	1.185170
0.12	1.189048	1.192205	1.195318	1.198394	1.201441	1.204465	1.207470	1.210461	1.213439	1.216410	1.219376
0.13	1.220947	1.224613	1.228230	1.231807	1.235353	1.238874	1.242376	1.245865	1.249343	1.252816	1.256285
0.14	1.255166	1.259371	1.263523	1.267633	1.271711	1.275764	1.279800	1.283822	1.287838	1.291851	1.295863
0.15	1.291674	1.296449	1.301168	1.305844	1.310487	1.315106	1.319710	1.324305	1.328896	1.333488	1.338084
0.16	1.330448	1.335824	1.341140	1.346414	1.351656	1.356876	1.362084	1.367288	1.372493	1.377705	1.382928
0.17	1.371471	1.377477	1.383422	1.389325	1.395199	1.401055	1.406905	1.412756	1.418616	1.424490	1.430382
0.18	1.414728	1.421393	1.427999	1.434563	1.441104	1.447633	1.454162	1.460700	1.467255	1.473834	1.480442
0.19	1.460212	1.467565	1.474861	1.482122	1.489364	1.496602	1.503850	1.511116	1.518411	1.525741	1.533111
0.20	1.507918	1.515991	1.524010	1.532000	1.539980	1.547966	1.555973	1.564010	1.572090	1.580217	1.588400
0.21	1.557849	1.566672	1.575448	1.584202	1.592958	1.601732	1.610540	1.619395	1.628306	1.637282	1.646330
0.22	1.610014	1.619619	1.629185	1.638741	1.648312	1.657917	1.667572	1.677290	1.687084	1.696961	1.706930
0.23	1.664427	1.674847	1.685239	1.695636	1.706063	1.716543	1.727092	1.737726	1.748455	1.759290	1.770238
0.24	1.721106	1.732377	1.743634	1.754912	1.766240	1.777642	1.789136	1.800738	1.812460	1.824312	1.836303
0.25	1.780080	1.792238	1.804400	1.816603	1.828880	1.841254	1.853748	1.866375	1.879151	1.892085	1.905185
0.26	1.841381	1.854465	1.867575	1.880751	1.894027	1.907428	1.920979	1.934694	1.948588	1.962672	1.976953
0.27	1.905048	1.919102	1.933207	1.947405	1.961734	1.976222	1.990892	2.005761	2.020844	2.036152	2.051692
0.28	1.971129	1.986199	2.001348	2.016625	2.032066	2.047703	2.063560	2.079655	2.096003	2.112614	2.129495
0.29	2.039679	2.055815	2.072063	2.088477	2.105095	2.121950	2.139068	2.156467	2.174161	2.192161	2.210474
0.30	2.110759	2.128016	2.145425	2.163041	2.180906	2.199055	2.217513	2.236300	2.255430	2.274913	2.294753
0.31	2.184442	2.202881	2.221516	2.240405	2.259595	2.279119	2.299006	2.319274	2.339937	2.361003	2.382477
0.32	2.260809	2.280496	2.300430	2.320672	2.341270	2.362262	2.383673	2.405523	2.427824	2.450585	2.473806
0.33	2.339949	2.360959	2.382273	2.403955	2.426056	2.448613	2.471654	2.495197	2.519253	2.543828	2.568923
0.34	2.421965	2.444380	2.467163	2.490383	2.514090	2.538324	2.563111	2.588468	2.614407	2.640929	2.668033
0.35	2.506968	2.530881	2.555234	2.580098	2.605527	2.631559	2.658220	2.685528	2.713488	2.742103	2.771366
0.36	2.595085	2.620598	2.646633	2.673262	2.700540	2.728506	2.757184	2.786589	2.816726	2.847593	2.879180

TABLE III.—CORRECTION FACTOR FOR THE SECOND OVERTONE OF FLEXURAL VIBRATION, T_3, AS A FUNCTION OF THE DIAMETER TO LENGTH RATIO d/l, AND POISSON'S RATIO μ.

Diameter to Length Ratio, d/l	Poisson's Ratio, μ										
	0.00	0.05	0.10	0.15	0.20	0.25	0.30	0.35	0.40	0.45	0.50
0.00	1.000000	1.000000	1.000000	1.000000	1.000000	1.000000	1.000000	1.000000	1.000000	1.000000	1.000000
0.01	1.002609	1.002660	1.002710	1.002759	1.002808	1.002856	1.002903	1.002950	1.002997	1.003043	1.003089
0.02	1.010421	1.010623	1.010823	1.011020	1.011214	1.011405	1.011594	1.011780	1.011968	1.012152	1.012335
0.03	1.023390	1.023845	1.024290	1.024729	1.025164	1.025593	1.026015	1.026436	1.026855	1.027267	1.027676
0.04	1.041446	1.042248	1.043034	1.043810	1.044578	1.045334	1.046082	1.046824	1.047563	1.048293	1.049019
0.05	1.064495	1.065736	1.066954	1.068155	1.069343	1.070516	1.071678	1.072830	1.073974	1.075110	1.076240
0.06	1.092436	1.094201	1.095938	1.097648	1.099340	1.101014	1.102673	1.104318	1.105953	1.107578	1.109196
0.07	1.125153	1.127523	1.129857	1.132160	1.134438	1.136693	1.138930	1.141151	1.143360	1.145557	1.147746
0.08	1.162526	1.165581	1.168592	1.171565	1.174505	1.177421	1.180314	1.183191	1.186053	1.188904	1.191747
0.09	1.204448	1.208262	1.212021	1.215737	1.219416	1.223067	1.226696	1.230306	1.233902	1.237488	1.241067
0.10	1.250814	1.255455	1.260034	1.264564	1.269056	1.273517	1.277955	1.282376	1.286786	1.291188	1.295587
0.11	1.301530	1.307065	1.312532	1.317947	1.323321	1.328666	1.333990	1.339300	1.344603	1.349904	1.355208
0.12	1.356516	1.363010	1.369431	1.375799	1.382127	1.388429	1.394714	1.400992	1.407270	1.413554	1.419850
0.13	1.415707	1.423222	1.430664	1.438052	1.445405	1.452737	1.460061	1.467387	1.474723	1.482077	1.489456
0.14	1.479051	1.487650	1.496175	1.504653	1.513102	1.521541	1.529982	1.538439	1.546921	1.555435	1.563990
0.15	1.546513	1.556257	1.565933	1.575568	1.585187	1.594809	1.604449	1.614123	1.623839	1.633609	1.643439
0.16	1.618072	1.629024	1.639915	1.650780	1.661643	1.672528	1.683453	1.694432	1.705478	1.716601	1.727810
0.17	1.693723	1.705945	1.718120	1.730286	1.742473	1.754704	1.767001	1.779381	1.791856	1.804437	1.817134
0.18	1.773475	1.787033	1.800562	1.814105	1.827696	1.841361	1.855124	1.869002	1.883011	1.897161	1.911461
0.19	1.857353	1.872312	1.887269	1.902268	1.917349	1.932540	1.947868	1.963350	1.979003	1.994838	2.010865
0.20	1.945396	1.961827	1.978286	1.994826	2.011487	2.028302	2.045298	2.062497	2.079912	2.097557	2.115440
0.21	2.037655	2.055631	2.073675	2.091843	2.110181	2.128725	2.147502	2.166535	2.185839	2.205426	2.225305
0.22	2.134200	2.153799	2.173513	2.193404	2.213523	2.233906	2.254584	2.275579	2.296906	2.318578	2.340601
0.23	2.235112	2.256416	2.277894	2.299610	2.321620	2.343962	2.366670	2.389764	2.413260	2.437169	2.461496
0.24	2.340488	2.363589	2.386928	2.410580	2.434600	2.459032	2.483908	2.509250	2.535071	2.561381	2.588182
0.25	2.450441	2.475435	2.500745	2.526451	2.552613	2.579275	2.606470	2.634219	2.662535	2.691423	2.720883
0.26	2.565101	2.592092	2.619491	2.647382	2.675827	2.704873	2.734552	2.764882	2.795876	2.827534	2.859853

The value of T_n for the assumed value of μ is then given by

$$T_n(x, \mu) = \frac{T_n(x, \mu_2) - T_n(x, \mu_1)}{\mu_2 - \mu_1} \cdot (\mu - \mu_1) + T_n(x, \mu_1) \quad \ldots\ldots(7)$$

A first value of E can now be computed from the appropriate one of Eqs 2, 3, or 4. If G is known, a more accurate value of μ can now be obtained, and substituted into Eq 7 for a better value of T_n, and hence E. This process should be repeated until there is no further change in the values of E and μ.

Interpolation by this method gives an accuracy in the T_n values of one part in 10^4 or better over the range of the tables. If higher accuracy is needed, Eqs 5, 6, and 7 should be replaced by Aitken's interpolation formulae (4).

Prisms of Rectangular Cross-Section:

The best existing theory for the flexural vibration of prisms is that of Pickett (5). For the fundamental mode of vibration, his equation is equivalent to

$$E = 0.94642 \frac{\rho l^4 f^2}{t^2} T \quad \ldots\ldots\ldots(8)$$

where T is a correction factor given approximately (6) by:

$$T = 1 + 6.585(1 + 0.0752\,\mu + 0.8109\,\mu^2)\left(\frac{t}{l}\right)^2 - 0.868\left(\frac{t}{l}\right)^4 - \frac{8.340\,(1 + 0.2023\,\mu + 2.173\mu^2)\left(\frac{t}{l}\right)^4}{1 + 6.338(1 + 0.14081\mu + 1.536\mu^2)\left(\frac{t}{l}\right)^2} \quad .(9)$$

Equation 9 is very accurate for long, thin specimens, but the error increases with increasing t/l. For very thick specimens ($t/l \approx 0.4$) the error is estimated to be about 1 per cent (6).

Because of the approximate nature of Pickett's theory, the use of overtones is not recommended for rectangular specimens.

LONGITUDINAL RESONANCE

Longitudinal resonances of cylindrical rods or bars of square cross-section are recommended as the most accurate method of determining Young's modulus. In the case of cylindrical specimens, the theory has been thoroughly worked out. Although the theoretical treatment is not nearly so adequate for square specimens, an empirical investigation (7) has revealed that the existing theory for cylinders may, with certain assumptions, be applied to specimens of square cross-section with a high degree of accuracy.

Cylindrical Specimens:

An approximate solution for the velocity of longitudinal waves in an infinitely long rod as a function of the diameter to wavelength ratio d/λ was derived by Rayleigh (8). A more recent paper by Bancroft (9) gives an accurate numerical solution of this same problem. These theories may be applied to resonance vibrations under the assumption that the vibrating rod contains an integral number of half wavelengths; that is, that

$$\lambda = \frac{2l}{n} \quad \ldots\ldots\ldots\ldots\ldots(10)$$

With this assumption, the equation for computing Young's modulus is:

$$E = \frac{\rho}{K_n}\left(\frac{2l f_n}{n}\right)^2 \quad \ldots\ldots\ldots\ldots(11)$$

where K_n is the correction factor for the nth mode of longitudinal vibration.

Rayleigh's correction factor, valid for the case $d/\lambda \ll 1$, may be expressed as:

$$K_n \approx 1 - \frac{\pi^2 n^2 \mu^2 d^2}{8 l^2} \quad \ldots\ldots\ldots(12)$$

If the correction term in Eq 12 is not small compared to 1, then Bancroft's results, given in Table IV, must be used. In order to use Table IV, compute λ from Eq 10 and interpolate K_n linearly with respect to $(d/\lambda)^2$ and μ, as described above for the section on flexural resonance of cylinders. This interpolation should be accurate to about 0.03 per cent. For greater accuracy, Aitken's interpolation method should be used.

TABLE IV.—THE CORRECTION FACTOR, K_n, FOR LONGITUDINAL RESONANCE VIBRATIONS OF CYLINDERS, AS A FUNCTION OF POISSON'S RATIO, μ, AND THE DIAMETER TO WAVELENGTH RATIO, d/λ.

λ is related to the length of the specimen, l, by the equation $\lambda = \frac{2l}{n}$, where $n = 1, 2, \cdots$.

Diameter to Wavelength Ratio, d/λ	Poisson's Ratio, μ						
	0.10	0.15	0.20	0.25	0.30	0.35	0.40
0.00	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000
0.05	0.99988	0.99972	0.99950	0.99922	0.99888	0.99848	0.99802
0.10	0.99950	0.99886	0.99798	0.99686	0.99549	0.99389	0.99206
0.15	0.99882	0.99736	0.99533	0.99277	0.98967	0.98609	0.98202
0.20	0.99780	0.99509	0.99138	0.98670	0.98117	0.97480	0.96772
0.25	0.99632	0.99184	0.98579	0.97830	0.96956	0.95975	0.94903
0.30	0.99421	0.98728	0.97810	0.96702	0.95435	0.94049	0.92571
0.35	0.99114	0.98085	0.96759	0.95203	0.93486	0.91660	0.89770
0.40	0.98651	0.97158	0.95310	0.93236	0.91031	0.88770	0.86503
0.45	0.97913	0.95778	0.93300	0.90668	0.88003	0.85372	0.82812
0.50	0.96621	0.93646	0.90503	0.87383	0.84372	0.81499	0.78780

Prisms of Square Cross-Section:

The theory given above for longitudinal resonance in cylindrical specimens may be applied to specimens of square cross-section under the assumption that a square specimen of width b and length l behaves exactly like a cylindrical specimen of diameter d and length l, if d and b are related by the equation

$$d^2 = \frac{4}{3} b^2 \qquad \text{(13)}$$

This assumption is reasonable since it merely requires that the moment of cross-sectional area about an axis through the center and along the length of the specimen be the same for the square as for the equivalent cylindrical specimen. Furthermore, this relationship has been found to hold experimentally to a very high degree of accuracy (7).

Thus, to compute Young's modulus from the longitudinal resonance frequencies of square specimens, calculate K_n from Table IV (or Eq 12 if applicable) using the relations shown in Eqs 10 and 13, and substitute this value of K_n into Eq 11.

Prisms of Rectangular Cross-Section:

The theory of longitudinal resonance vibrations of rectangular specimens has not been adequately determined, and good empirical relationships are unavailable. However, if the cross-section does not depart too greatly from square, then the methods given for longitudinal resonances of prisms of square cross-section may be used without introducing appreciable error if the following equation is substituted for Eq 13:

$$d^2 = \frac{2}{3} (a^2 + b^2) \qquad \text{(14)}$$

If the cross-section is not approximately square, the errors involved in the use of Eq 14 will increase. In this case, the use of longitudinal resonance vibrations is not recommended since Young's modulus may then be computed more accurately and easily from flexural resonance.

TORSIONAL RESONANCE

It was mentioned previously that torsional vibrations are important not only because a knowledge of the shear modulus might be desirable, but also because an accurate determination of Young's modulus requires a knowledge of Poisson's ratio, which is in turn a function of E/G.

The theory of this type of vibration is both accurate and simple for cylindrical specimens. However, this is also the shape for which experimental difficulties are formidable. Prismatic specimens, on the other hand, are the easiest to deal with experimentally, but the equations involved are neither as simple nor as accurate as those for cylinders. One is therefore forced to choose between experimental difficulty and accuracy.

The basic equation for torsional resonance is:

$$G = \rho \left(\frac{2l f_n}{n} \right)^2 R \quad \ldots\ldots\ldots (15)$$

where R is a complicated function of the shape of the specimen.

Cylindrical Specimens:

The shape factor R is exactly 1 for cylinders, independent of the value of d/l. Equation 15 is therefore exact for this shape specimen, with the substitution[7] of

$$R = 1 \quad \ldots\ldots\ldots\ldots\ldots (16)$$

Prisms of Square Cross-Section:

Davies (10) derived approximate equations for the torsional resonance of rectangular bars. Although these equations are in qualitative agreement with experiment in the special case of bars of square cross-section, more accurate results (11) can be obtained with the empirical Eq 11

$$R = R_0 \left[1 + n^2 (b/l)^3 (0.01746 + 0.00148n + 0.00009n^2)\right] \quad \ldots (17)$$

where R is the shape factor of Eq 15 and R_0 is the shape factor for infinitely thin, square specimens:

$$R_0 = 1.18559 \quad \ldots\ldots\ldots\ldots (18)$$

Equation 17 is believed to be accurate to within 0.01 per cent for any combination of n and b/l which can be reasonably obtained experimentally.

Prisms of Rectangular Cross-Section:

An accurate theory is not available for the torsional resonance of rectangular bars. Experimental considerations, however, frequently force one to use this shape, so the best approximations available will be given.

Timoshenko (12) solved the equations for static torsion of rectangular specimens. His results are given in the form of an infinite series, which may be expressed to a high degree of accuracy by the equation:

$$R_0 = \frac{1 + \left(\frac{b}{a}\right)^2}{4 - 2.521 \frac{a}{b} \left(1 - \frac{1.991}{e^{\pi \frac{b}{a}} + 1}\right)} \quad \ldots\ldots (19)$$

Equation 19 reduces to the value given

---

[7] It is clear from Eqs 15, 16, 11, and 1 that if the cross-sectional correction K_n is neglected in Eq 11, then for cylindrical specimens μ may be computed directly from the fundamental resonance frequencies from the relation:

$$\mu = \tfrac{1}{2} \left(\frac{f_l}{f_t} \right)^2 - 1$$

where f_l is the fundamental longitudinal resonance frequency and f_t is the fundamental torsional resonance frequency.

in Eq 18 for the particular case $a = b$, that is, for a square cross-section.

For the case $a \ll b$, this equation can be approximated by an equation similar to that given by Pickett (5):

$$R_0 \approx \frac{1 + \left(\frac{b}{a}\right)^2}{4 - 2.521\,\frac{a}{b}} \quad \ldots\ldots\ldots (20)$$

In an empirical investigation, Spinner and Valore (13) found that for $b/l \approx 0.21$ and $1 \leq b/a \leq 10$, the value of R for the dynamic case differed somewhat from Pickett's equation. A combination of Davies' theoretical results and Spinner's empirical results can probably be best expressed by the equation:

$$R = \left[\frac{1 + \left(\frac{b}{a}\right)^2}{4 - 2.521\,\frac{a}{b}\left(1 - \frac{1.991}{e^{\pi\frac{b}{a}} + 1}\right)}\right] \cdot \left[1 + \frac{0.00851\, n^2 b^2}{l^2}\right] - 0.060\left(\frac{n\,b}{l}\right)^{\frac{3}{2}}\left(\frac{b}{a} - 1\right)^2 \quad \ldots\ldots (21)$$

for the shape factor R of Eq 15.

Equation 21 should be accurate to within about 0.2 per cent for $b/l \lesssim 0.3$ and $b/a \lesssim 10$, in the fundamental mode of vibration. It is difficult to estimate the expected errors for higher values of b/l, b/a, or n, since sufficient data to show the effects of these three parameters are not yet available. It is estimated, however, that except for extreme cases, the errors should not exceed 1 per cent.

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DISCUSSION

MR. R. E. PHILLEO.[1]—The authors indicate a very high degree of accuracy in frequency determination. How is the oscillator set to give this degree of accuracy? Do you merely observe the oscilloscope pattern or do you employ an auxiliary galvanometer?

Has any quantitative work been done on damping?

MR. W. E. TEFFT (*author*).—All of the materials used in the work referred to here have had low damping factors. Since we have also taken great care to reduce the damping due to the suspension, the result has been a very sharp response at resonance. Under such conditions observation of the oscilloscope pattern is quite adequate for accurately determining resonance. In fact, the accuracy of frequency determination has usually been limited primarily by the addition of stiffness to the system by the suspension.

These two causes of error can easily be distinguished in practice, since the first gives random variations in measured frequency with a fixed suspension position, while the second gives different values only when the suspension is changed.

In answer to the second question, this technique has been used successfully for internal friction measurements. Some of the precautions to be taken, and limitations of the method are described in an article by J B. Wachtman, Jr. and W. E. Tefft, *Review of Scientific Instruments*, Vol. 29, p. 517 (1958).

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[1] Office of Chief of Engineers, Civil Works, Corps of Engineers, Washington, D. C.

SOME OBSERVATIONS ON THE MEASUREMENT OF SENSITIVITY OF CLAYS*

By W. J. Eden[1] and J. K. Kubota[2]

Synopsis

Sensitivity is based on actual measurements of the undrained shear strength of a clay. It should accordingly be a better criterion to describe soil properties than measures such as the liquidity index. No critical values of sensitivity have appeared, however, that would distinguish a clay subject to earthflows from one that is not. It may be that the present methods of measuring sensitivity lack the necessary precision.

In this paper, four methods of measuring sensitivity, the field vane, the unconfined compression test, the laboratory vane, and the fall-cone test are described in some detail. A comparison is made between the results of the four methods in the light of observations made on a number of borings in Leda clay. The sensitivities ranged from 1 to over 300. It was found that there was considerable discrepancy between the results obtained by the four methods.

Sensitivity in soils is defined as the ratio of the undisturbed strength to the remolded strength at the same water content. It is usual to report values of sensitivity along with other geotechnical properties of clay. Suggestions have been made by Skempton and Northey (**1**)[3] and Bjerrum (**2**) to classify clays by their sensitivity; Bjerrum shows a general relationship between the liquidity index $\left(LI = \dfrac{WC - PL}{LL - PL}\right)$ and the sensitivity. As yet no critical values of sensitivity have appeared, that would distinguish a clay subject to flow slides from one that is not. As sensitivity is based on actual measures of shear strength, logically it should be a better criterion to describe soil properties than the liquidity index. This may not be possible because of the wide variations of sensitivity in a single clay profile, but the authors suggest that another reason may be that methods now used to obtain a measure of sensitivity lack the necessary precision.

Terzaghi and Peck (**3**) suggest that sensitivity be defined as the ratio of the undisturbed unconfined compression strength to the remolded unconfined compressive strength at the same water content. This method is satisfactory for clays of relatively low sensitivities, but as the liquidity index of the clay approaches 1, it is no longer possible to form remolded specimens that have enough strength to be self-supporting for an unconfined compression test. Thus for clays with high sensitivities, alternate means of measuring the sensitivity had to be sought. In the literature, values of sensitivity are reported based on (1) field vane tests, (2) laboratory vane tests, and (3) laboratory fall-cone tests.

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[2] Professor of Civil Engineering, Kobe University, Kobe, Japan. Visiting professor with the Division of Building Research, National Research Council, Ottawa, Canada, during the summer of 1960.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

Andresen and Bjerrum (4) report on the measurement of sensitivity with the field vane test. Their method was to remold the soil by rotating the vane rapidly for 25 revolutions, then taking the remolded strength without allowing the rotation to stop. Their apparatus completely eliminates friction between the rods connecting the vane to the surface from the surrounding soil and can be rotated quite rapidly. Eden and Hamilton (5) found that sensitivities measured by another field vane were not reliable. With the latter apparatus, friction between the soil and the rod was a significant factor in remolded strength determinations, and the vane was only rotated four times rather slowly to remold the soil, and there was a delay of about 1 min between the completion of the remolding and the start of the test. Even with the better quality equipment, Andresen and Bjerrum (4) report that the sensitivity values obtained with the field vane test are somewhat less than measured in the laboratory.

The laboratory vane test follows the same principle as that of the field vane test in that a 4-bladed vane is inserted into a soil specimen, and the torque necessary to rotate the vane is measured and related to the undrained strength. It has been used on both undisturbed and remolded clay samples to measure the sensitivity. In practice, it can be used to measure sensitivity over a fairly wide range, but when the remolded strength becomes extremely small, it is difficult to measure torque with enough discrimination to give reliable sensitivity values.

In the fall-cone test, a cone of known weight and dimensions is brought into contact with the surface of the clay. It is released for about a 5-sec interval and allowed to penetrate the clay under its own weight. The penetration is then measured and if desired related to shear strength as suggested by Hansbo (6) by $S_s = (kQ)/(H^2)$ where S_s is shear strength, Q the weight of the cone, H is the penetration, and k a cone constant. The test can be done on both undisturbed and remolded samples. The sensitivity is then defined by (H^2 remolded)/(H^2 undisturbed). The cone is useful over a limited range of sensitivity. For the undisturbed test, the penetration should be at least 5 mm to be reliable, and if the remolded strength is very low, the cone buries itself in the soil.

Hansbo (6) reports that the sensitivity as determined by the fall-cone test was taken simply as (H remolded)/(H undisturbed) although this was not in full accordance with the definition of sensitivity.

In the present study, the four described methods of measuring sensitivity are compared. In addition, the sensitivity as measured by the ratio of one half the unconfined compressive strength to the remolded laboratory vane strength is compared with the first four methods.

BORINGS USED FOR COMPARISON

To compare the results of the four test methods, the measured sensitivity from four borings in Leda clay in the Ottawa area are presented. All four borings are located within 20 miles of Ottawa so that the general description of the clay given by Crawford (7) applies.

The first boring is located on the eastern outskirts of Ottawa on the Montreal Road property of the National Research Council. The total depth of the clay at this site is about 60 ft with a fissured crust extending to a depth of at least 30 ft. The surface elevation is about 310. The clay is extremely sensitive and highly stratified at depths below 40 ft.

The salt concentration of the pore water is less than 1 g per liter at this site.

The second boring was located about two miles east of the first boring on a lower terrace of the Leda clay. The surface elevation is about 180, and the depth of clay is about 120 ft. The pore water salt concentration at this boring is from 5 to 13 g per liter, and the clay is much less sensitive than in the first boring.

The third boring is located on the same terrace as the second boring, but about one mile north near the bank of the Ottawa River. The salt concentration is less than 2 g per liter and the sensitivity much higher than in the second boring. The surface elevation is 175 and the clay extends to a depth of nearly 100 ft.

The final boring is located about 20 miles south of Ottawa near the village of Kars. The boring is situated beside the Rideau River. The clay extends to 50 ft, the top 20 ft being fissured. From 20 to 40 ft the clay has a very low salt concentration and is extremely sensitive.

In addition, some detailed tests were conducted on two block samples obtained from deep excavations in the city of Ottawa.

The test methods used to compute sensitivity, S_t, are numbered 1 to 5 as follows:

$$\text{No. 1 } S_t = \frac{\text{half the unconfined compressive strength}}{\text{remolded laboratory vane strength}}$$

$$\text{No. 2 } S_t = \frac{\text{undisturbed laboratory vane strength}}{\text{remolded laboratory vane strength}}$$

$$\text{No. 3 } S_t = \frac{(\text{penetration of fall cone on remolded soil})^2}{(\text{penetration of fall cone on undisturbed soil})^2}$$

$$\text{No. 4 } S_t = \frac{\text{undisturbed field vane strength}}{\text{remolded field vane strength}}$$

$$\text{No. 5 } S_t = \frac{\text{penetration of fall-cone on remolded soil}}{\text{penetration of fall-cone on undisturbed soil}}$$

APPARATUS

Field Vane Apparatus.—In three of the four borings, the field vane manufactured by Geonor, Oslo, Norway, was used. This apparatus is described in detail by Andresen and Bjerrum (4) and their procedure was followed. The only modification was that where the undisturbed strength exceeded 2000 lb per sq ft, a 43 by 86-mm vane was used in place of the 55 by 110-mm vane. This reduced the discrimination of the torque measuring equipment somewhat.

Laboratory Vane Apparatus.—This apparatus (Fig. 1) was mounted on a Soiltest triaxial frame. The vane itself was $1\frac{1}{4}$ by $\frac{3}{4}$ in. and patterned after that of Skempton and Bishop (8). The torque was measured by a dynamometer ring acting through a lever system. The variable speed drive on the Soiltest triaxial frame was utilized to provide a nearly constant rate of rotation for the vane.

For the undisturbed test, a cylinder of soil, $2\frac{1}{4}$ in. in diameter by 3 in. long, was wrapped tightly in metal foil and sealed in wax to restrain the specimen laterally. The retainer was firmly seated in the bottom of the specimen. The top surface was cleaned, and the sample was thrust upwards so the vane was buried at the middle of the specimen. Torque was applied so that the vane was turned at about 6 deg per min. It has been found that the method of wrapping and holding the specimens was satisfactory for soft clays. For firm and stiff clays, however, the insertion of the vane sometimes split the specimen; hence the undisturbed laboratory vane is generally restricted to use in soft clays. In these clays the strength thus obtained generally agrees well with one half the unconfined compression strength.

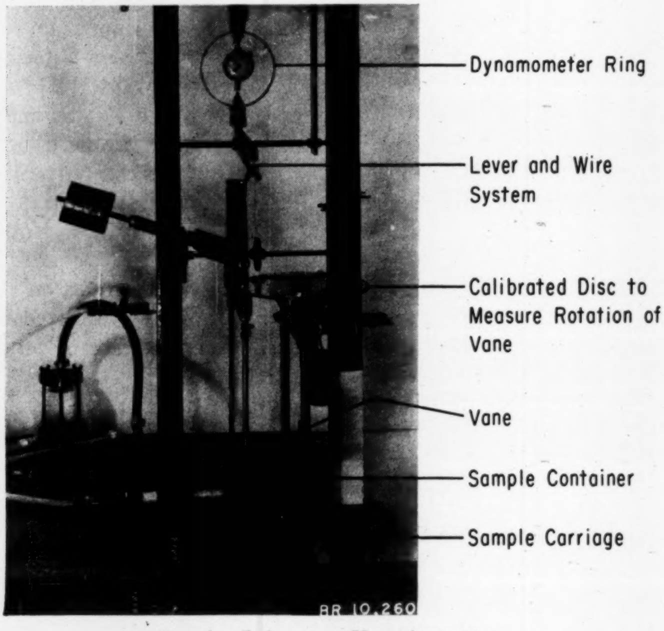

FIG. 1.—Laboratory Vane Apparatus.

After the undisturbed test, the specimen was remolded thoroughly with a mechanical mixer. The resulting slurry was deposited in a Lucite container $2\frac{3}{4}$ in. in diameter by 3 in. long. The vane was thrust into the slurry to determine the remolded strength at the same rate of strain as the undisturbed determination. A period of 1 min elapsed between the completion of the remolding process and the start of torque application.

At first, a dynamometer ring with a capacity of 50 lb was used. It was soon found that this ring did not give sufficient discrimination for remolded clays with a very low shear strength. A second special ring with a capacity of 15 lb was made, which could be interchanged with the 50-lb ring. With this ring, it is possible to measure shear strengths as low as 0.02 psi.

Fall-Cone Apparatus.—This was purchased from Geonor, Oslo, Norway. It consists of an instrument head mounted on a vertical arm free to be moved up and down the arm by a rack and pinion arrangement. The cone is held in position by a small electromagnet. It falls freely when the current supply to the electromagnet is switched off. The penetration of the cone is measured by observing a scribed mark on the cone through an optical scale.

Three cones were used with the apparatus regularly, a 30-deg cone weighing 100 g, a 60-deg cone weighing 60 g and a 60-deg cone weighing 10 g. The choice of cone depends on the consistency of the undisturbed sample. A penetration of at least 5 mm was desirable to obtain a reasonable assessment of the strength. It was preferable to use the same cone for the remolded strength determination, but if penetration in the remolded clay was too great, a lighter cone was substituted. The cone readings were then compared by Hansbo's equation:

$$S_s = (kQ)/(H^2) \ldots\ldots\ldots\ldots (1)$$

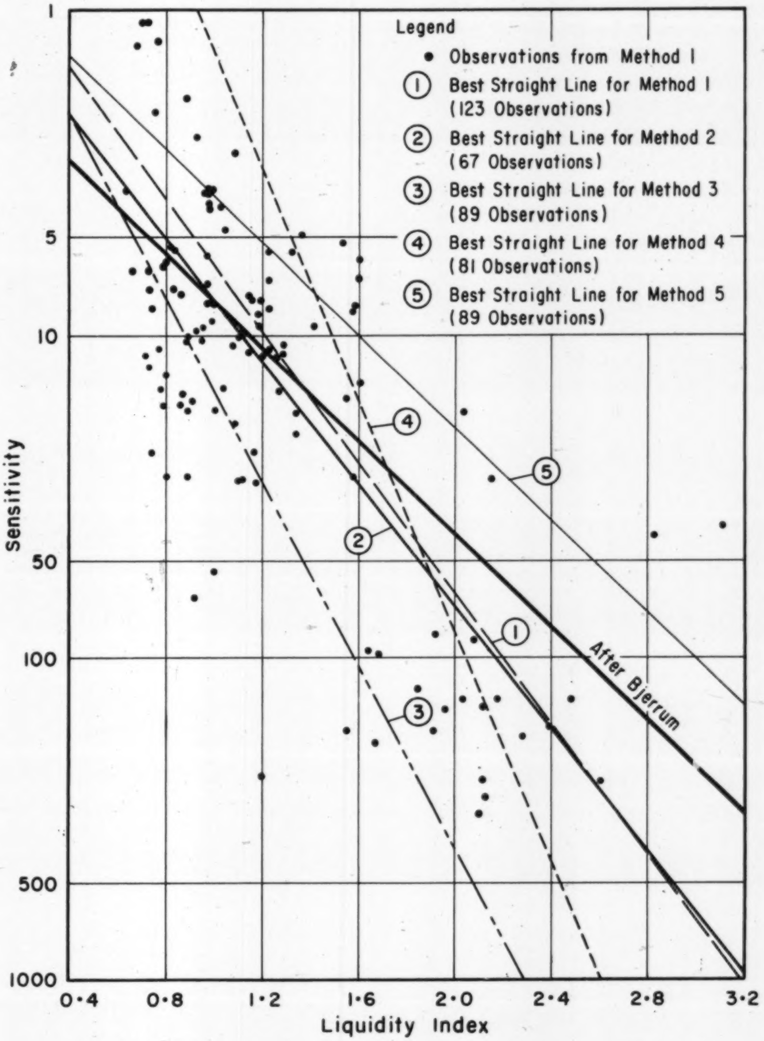

FIG. 2.—Relation Between Liquidity Index and Sensitivity by Five Methods.

The cone readings were repeated at least three times for each determination to rule out effects of the cone encountering a sand grain or other obstruction.

TREATMENT OF RESULTS

Bjerrum (2) shows a logarithmic correlation between sensitivity and liquidity index. This correlation has been used for the comparison of the five methods of measuring sensitivity. Figure 2 shows the individual observations for method No. 1 along with the best straight lines for the other four methods. The correlation established by Bjerrum is also shown.

In general, the Leda clays appear to be more sensitive for the same liquidity index than the Norwegian clays. This may be because Leda clays generally have a higher plasticity index than the Norwegian clays, and hence, an excess water content over the liquid limit, say of 5 per cent, leads to higher values of liquidity index in the case of the Norwegian clays. This feature is shown in Fig. 3, the activity chart comparing the two clays.

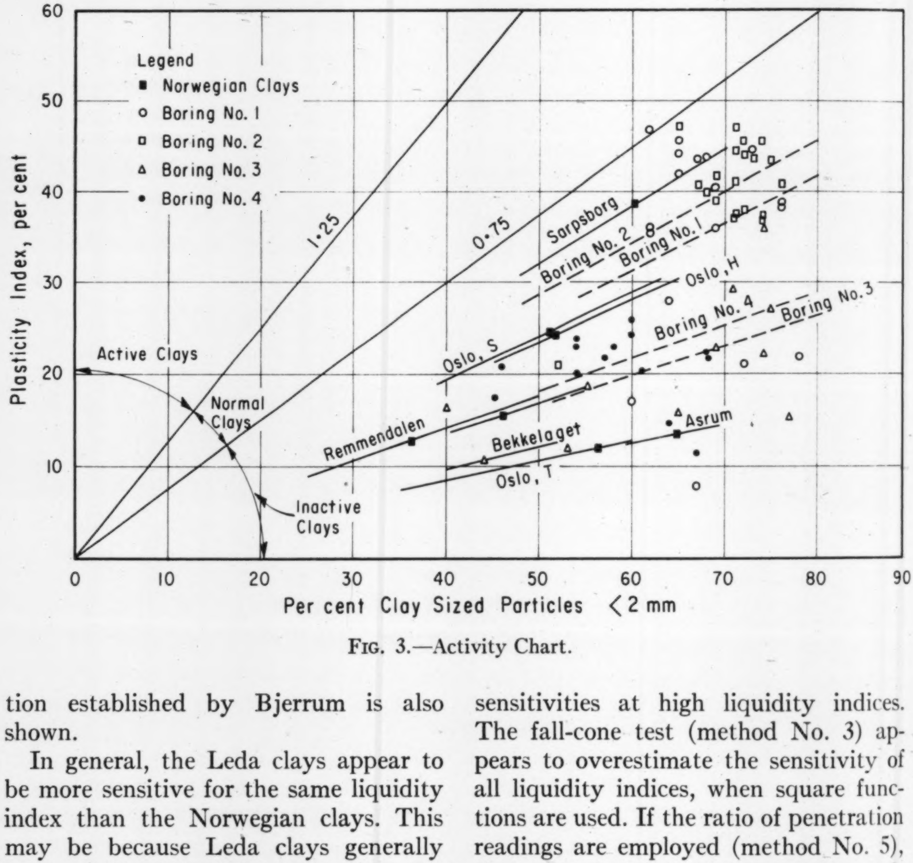

FIG. 3.—Activity Chart.

Of the five methods shown in Fig. 2, the laboratory vane and unconfined methods (methods Nos. 2 and 1) present the best agreement with Bjerrum's relationship. If Bjerrum's correlation is valid for Leda clay, the field vane method (No. 4) tends to underestimate the sensitivity when the liquidity index is less than 1.5 and overestimate the sensitivities at high liquidity indices. The fall-cone test (method No. 3) appears to overestimate the sensitivity of all liquidity indices, when square functions are used. If the ratio of penetration readings are employed (method No. 5), then the sensitivity is underestimated.

It will be noted that the results presented for the one test method are badly scattered. The results of the other four methods exhibit a similar scatter and have not been shown on Fig. 2. The scatter may be attributed to the fact that neither the liquidity indices nor the sensitivity values are precise measurements.

The liquidity indices used in the correlation are the average values of a 2-ft layer of clay. The sensitivity measurements were made on samples representing at a maximum a 3-in. layer of clay. Thus the true liquidity index for the 3-in. layer could have been significantly different from the value obtained for a 2-ft layer. To illustrate this point, in one sample of stratified clay the liquidity index varied from 1.3 to 2.3 in a 3-ft length, with the average being 2.0 for the total length. In general, if the liquidity index was high, larger variations could be expected.

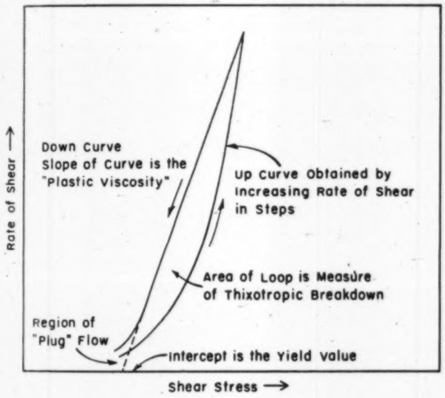
Down Curve
Slope of Curve is the "Plastic Viscosity"
Up Curve Obtained by Increasing Rate of Shear in Steps
Area of Loop is Measure of Thixotropic Breakdown
Region of "Plug" Flow
Intercept is the Yield Value
Rate of Shear →
Shear Stress →

FIG. 4.—Consistency Curve for Thixotropic Plastic Material. (After Green (9).)

All methods of measuring sensitivity suffer from lack of precision in determining the remolded strength. This is especially true when the sensitivity is high, and it may be stated generally that the higher the sensitivity, the less reliable it is likely to be.

GENERAL DISCUSSION

The question now arises as to the preferred method of measuring sensitivity. For the purposes of this argument, it is assumed that all methods can be depended upon to assess the undisturbed strength reliably. Thus the discussion will center around the application of the four test methods for measuring the remolded strength.

Since a clay slurry may be compared with paint or printing ink in consistency, it may be useful to examine the methods used to measure the viscous properties of paint. Green (9) has shown a consistency curve as determined by a rotational viscometer for a thixotropic plastic suspension, the features of which are shown in Fig. 4. If shear stress is plotted against rate of shear, suspensions of this class will demonstrate a yield value, that is, a measurable shear stress is necessary before any flow will take place. Once the yield stress is exceeded, flow will begin, and, as with all viscous materials, there is a relation between rate of shear and shear stress. If the material is thixotropic, then there is a hysteresis effect between ascending and descending rates of shear. Green (9) has suggested that the area defined by the hysteresis loop is a measure of thixotropy. The hysteresis loop can be determined with a rotational viscometer as described by Green. In a thixotropic material, the readings on the ascending run must be taken after a lapse of time to allow the set rate of shear to break down the thixotropic structure. For example, if the viscometer speed is set at 10 rpm at first a high shear strength will be measured which will decrease with time to a minimum value as the thixotropic structure is broken down. When the minimum value has been reached and recorded, the viscometer can be set to the next higher rate of shear. A further breakdown with time will occur at this higher rate of shear. The down curve is obtained without any influence from thixotropic breakdown.

At very low rates of shear, an additional complication is encountered, that of "plug flow." At slow angular velocities, the material moves around the bob of the viscometer as the cup rotates as a body without shearing within itself. The shearing action occurs in a thin zone either next to the wall of the cup or the bob, and hence it is necessary to extrapolate the down curve to obtain the yield value.

On a sample which had a sensitivity about 200, two tests were conducted on the remolded clay with a high-speed rotational viscometer. To prepare the specimens, the clay was remolded at its natural water content, then extruded through a 100 mesh screen to remove any sand particles which might influence the viscometer. The sample was then placed in the cup of the viscometer, the annular space between the bob and the cup being 2 mm. To prevent the sample from drying out during the test, it was necessary to cover the specimen with a thin film of mineral oil.

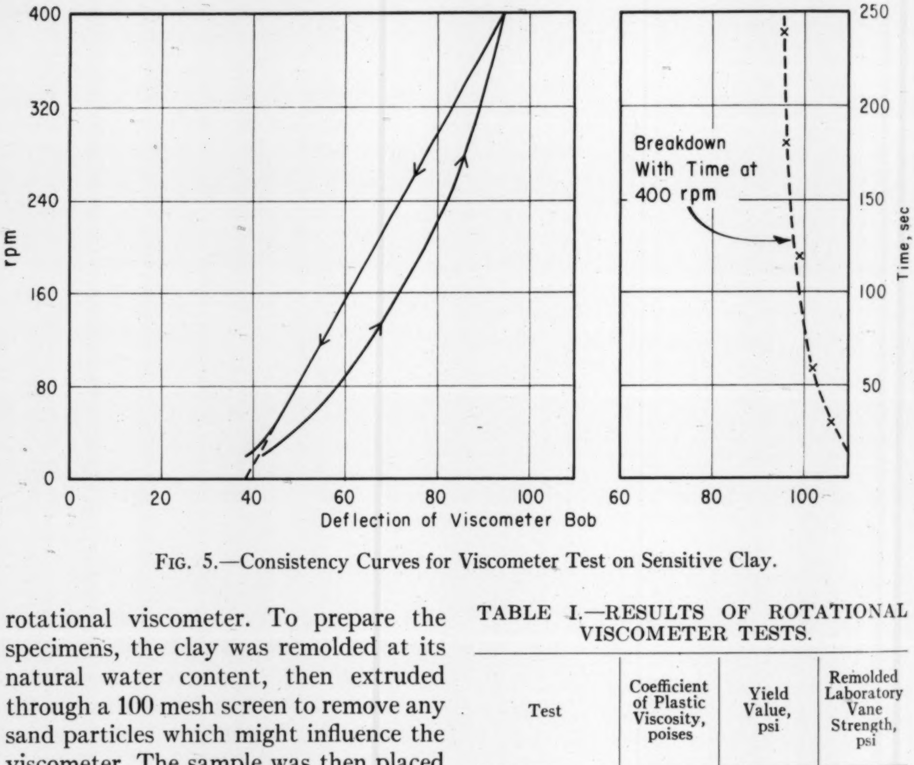

FIG. 5.—Consistency Curves for Viscometer Test on Sensitive Clay.

The cup of the viscometer was rotated at 20 rpm, this speed being maintained until the minimum stable reading was obtained. The speed was then increased to 40 rpm and the reading repeated.

In steps of 20 rpm, the speed was increased to 400 rpm. After the 400-rpm reading was obtained, it was necessary to stop the rotation because the sample was heating up. After the sample had cooled to the bath temperature, the cup was rotated once more at 400 rpm. A series of readings were taken to measure the breakdown with time at 400 rpm. When the deflection reading equalled that previously established at 400 rpm, the speed was decreased in steps of 20 rpm. For run No. 5, the steps were spaced 30 sec apart and number 6 run, 15 sec.

TABLE I.—RESULTS OF ROTATIONAL VISCOMETER TESTS.

Test	Coefficient of Plastic Viscosity, poises	Yield Value, psi	Remolded Laboratory Vane Strength, psi
67-21-5........	0.756	0.023	0.03
67-21-6........	0.763	0.021	0.03

The results of one viscometer run is presented in Fig. 5 along with the breakdown with time at 400 rpm. It will be apparent that the shape of curve obtained for the clay slurry resemble that of Green's hypothetical curve. Table I compares the values derived from the viscometer test in relation to the laboratory vane tests on the same remolded sample.

ASSESSMENT OF TEST METHODS

Considering now the methods of measuring remolded strength to derive their sensitivity, it may be argued that the field vane most closely duplicates the action of the viscometer. The remolding process is completed at a relatively high rate of shear, then readings are taken at a much lower rate of shear without a period of rest between remolding and reading. It is also believed that remolding action produces only a thin remolded surface to the order of 1 or 2 mm. This feature is illustrated in Fig. 6 which shows the surface of rupture around the field vane after a remolded test. The cylinder of soil retained in the blades of the vane in this instance was relatively undisturbed.

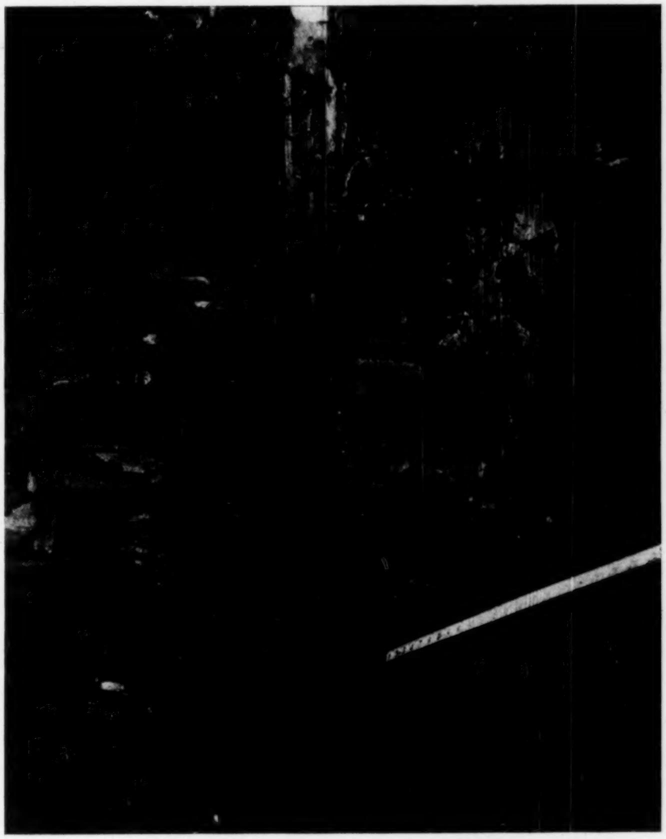

FIG. 6.—Remolded Surface Formed in Field Vane Test.

Another factor which may influence the field vane test is the pore water pressure and the rate with which it dissipates. Any loss of water during the remolding process because of drainage would lead to the remolded strength being too high.

With the laboratory vane test, the remolded clay is prepared in a container with a diameter much larger than that of the vane. With the more sensitive soils, the remolded clay is in a semi-fluid or fluid state. Under these conditions, it is questionable whether the shearing surface formed in the clay slurry is the cylinder conforming to the shape of the vane. If the soil tends to be thixotropic, the laboratory vane will yield too high a result because there is no breakdown process and the readings are taken during the ascending leg of the hysteresis loop.

As Fig. 2 has shown, the fall-cone test consistently yielded higher sensitivity values than the other test methods when the values of sensitivity were taken as (H^2 remolded)/(H^2 undisturbed). Hansbo has pointed out that for the same cone, the k value in $S_s = (kQ)/(H^2)$ is different for undisturbed and remolded clays. For example, considering the 60-deg, 60-g cone, k for undisturbed clay was approximately 0.25 for one type sampler and 0.20 for another. For remolded clay, the k for the 60-deg cone was about 0.30. Thus, in view of Hansbo's work, the simple comparison of the undisturbed and remolded cone penetrations can be expected to yield too high values of sensitivity.

CONCLUSIONS

If any conclusion can be drawn from this study, it is that the present methods of measuring sensitivity cannot be expected to yield consistent results. Not much significance can be attached to a single sensitivity measurement.

Factors that contribute to the rather poor results obtained at:

1. Insufficient sensitivity in apparatus for measuring the remolded strength,
2. Time interval between the completion of the remolding process and the start of the remolded strength determination, particularly in clays which are thixotropic, and
3. Whether or not the test is conducted with ascending or descending rates of shear.

The study has shown that no one method is to be preferred for the determination of the sensitivity. The field vane method is probably the most economical method, but it appears to underestimate sensitivity at low liquidity indices and overestimate it when the liquidity index is high. The fall-cone test is the most convenient and economical of the laboratory methods. The interpretation of the results of this test is open to doubt. If the function (H^2 remolded)/(H^2 undisturbed) is used as sensitivity, the results presented indicate that the sensitivities are higher than obtained with other methods. If (H remolded)/(H undisturbed) is used for the sensitivity the results are lower than obtained by the other methods.

It would appear that the Leda clays studied are slightly more sensitive for the same liquidity index than the relation suggested by Bjerrum.

In spite of the more or less negative findings resulting from this study, it is hoped that this paper will have made some contribution to the problem of measuring sensitivity and will assist in an appreciation of the sensitivities reported in the literature of soil mechanics.

Acknowledgments:

This paper is a contribution of the Division of Building Research, National Research Council of Canada, and is published with the approval of the Director of the Division.

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THE INFLUENCE OF STRAIN ON SHEARING RESISTANCE OF SENSITIVE CLAY*

BY CARL B. CRAWFORD[1]

SYNOPSIS

The effective stress shear strength parameters of a sensitive clay are subject to a wide degree of interpretation because the pore water pressure does not reach equilibrium at maximum stress. A simple method of obtaining shear strength in terms of fundamental parameters, believed to be equal to Hvorslev's true parameters, is described. A revised interpretation of true cohesion for undrained tests is developed. Water content variation during the test is discussed, and the mechanism of friction and cohesion is related to degree of deformation.

In accordance with the fundamental principle of effective stress, the shearing resistance on any plane in a soil depends not on the total stress but on the effective stress on the plane (**1**).[2] The effective stress is the so-called grain-to-grain stress found by subtracting the pore water pressure from the total stress. It is clear, therefore, that values for shear strength measured or applied without regard to pore water pressure may be subject to considerable error. Knowledge of this fact has created a great deal of interest in the measurement of shearing resistance in terms of effective stresses and the use of effective stresses in field application.

Although the use of effective stress analyses is a great advance over the total stress method, it is still an empirical approach dependent upon the accuracy of arbitrary tests, usually on very small specimens, in representing field conditions. Much of the study and application of effective stresses has been on remolded or compacted soils in which stress history and sensitivity may have little influence. Because extrapolation of this experience into the realm of natural soils may not be justified, a special study of the shear strength properties of undisturbed specimens of a sensitive clay is being carried out by the National Research Council of Canada, Division of Building Research. This paper describes a series of tests in which the effective stress shear strength parameters are subject to a wide range of interpretation, and an effort is made therefore to interpret the results in terms of fundamental parameters.

SHEAR STRENGTH PARAMETERS

Thirty years ago Terzaghi (**2**) showed that if two identical specimens of clay are brought to the same water content—one by simple compression, the other by additional compression followed by rebound—the former will be considerably stronger than the latter.

This important discovery that the stress history of a clay would have a considerable influence on its strength was followed by the classical work of Hvorslev (3) who systematically traced the variation in the shearing resistance of remolded clay as it was consolidated, rebounded, and recompressed. He was able, by relating shearing resistance to the "equivalent consolidation pressure" (that is, the pressure on the virgin consolidation curve corresponding to the void ratio at failure), to obtain an expression for shear strength in the form of two parameters which were unaffected by stress history. These two parameters, called the "effective angle of internal friction" and the "effective cohesion," represent a frictional component of shearing resistance which depends only on the effective stress on the plane of failure, and a cohesive component which depends only on the void ratio in the plane of failure. Although Hvorslev still prefers the original terms (4), they have become widely known as the "true angle of internal friction" and the "true cohesion" to distinguish them from empirical parameters in terms of effective stresses (5–7).

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* Presented at the Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

The measurement of the Hvorslev parameters is usually such an involved procedure, requiring a good supply of uniform material, that it has been carried out for only a very few natural soils. This is why engineers have continued to rely on empirical parameters determined in terms of total applied stress or in terms of applied effective stress in which a relationship is established between shearing resistance and effective stress over a wide range of water contents. The values obtained in terms of total stress are applicable only if no drainage occurs in the test or in the field problem. The values in terms of effective stress can account for long-term pore pressure changes, but if a substantial proportion of the shearing resistance of a soil actually consists of true cohesion the direct application of empirical parameters in terms of effective stresses may be of limited accuracy. In other words, as the difference between the Hvorslev true angle of internal friction and the angle of shearing resistance in terms of effective stress increases, the departure from reality becomes of greater significance.

SENSITIVE CLAYS

Sensitive clays present a special problem in the determination of effective stress parameters because the pore water pressure induced by shearing continues to increase after the maximum stress has been applied and the selection of the point of failure is controversial. This has led to a consideration of progressive shear strength parameter mobilization (8,9) and of failure criteria other than a maximum deviator stress. In a discussion submitted to the recent ASCE Research Conference on Shear Strength of Cohesive Soils (10), the author presented test results showing that for a sensitive marine clay the computed angle of shearing resistance, ϕ', increased with strain and was subject to variation of interpretation from 22 to 34 deg. Osterman (11) reported similar test results on some Swedish clays in which "angles of up to 41 deg were obtained for very soft clays, which cannot reasonably be correct." Although not a great deal of information is available, the variety of soils in which pore pressures continue to increase after the maximum deviator stress has been applied may be quite extensive. Parry (12) reports this phenomenon for the insensitive London clay.

PROPERTIES OF THE SOIL

The tests reported in this paper were made on samples of a late glacial marine clay called "Leda" clay. The properties of this clay, which occurs extensively in eastern Canada, have recently been summarized (13). The primary set of tests were performed on specimens cut from undisturbed block samples obtained from a small tunnel at a depth of 33 ft. The second set of tests are from a previously reported study of strain rate influences (**14**) and are quoted in support of the conclusions drawn from the primary tests.

TABLE I.—PROPERTIES OF THE SOIL.

Soil Properties	Specimens	
	96-4	83-27
Depth, ft	33	16
Elevation, ft	222	234
Natural water content, per cent	58	67
Liquid limit, per cent	53	65
Plastic limit, per cent	25	26
Specific gravity	2.80	2.80
Clay size, per cent	62	60
Salt in pore water, gm per l	2.0	0.5
Field vane strength, kg per sq cm	0.55	0.64
Sensitivity (approx)	50	20
Preconsolidation pressure, kg per sq cm	2.4	2.3

To reduce the scatter in test results, special efforts were made to obtain samples of nearly uniform water content, although this is not a general feature of the clay. Further, the samples were obtained from a depth believed to be below surface weathering effects. The characteristics of the two sets of samples, which were located about three miles apart in the City of Ottawa, are summarized in Table I.

TEST METHODS

Consolidated undrained triaxial tests were performed on specimens 10 sq cm in area and 8 cm high, wrapped in prepared

TABLE II.—TEST RESULTS.

Series[a]	Specimen	$\bar{\sigma}_c$[b]	$(\bar{\sigma}_1 - \bar{\sigma}_3)_f$[c]	T_f,[d] hr	Average Water Content, per cent		Water Content of Slices, per cent				
					Initial	Failure	1	2	3	4	5
A	96-4-1	2	1.26	1.0	59.4	51.7	...	...	...	...	...
	2	3	1.81	1.3	60.7	47.1	46.2	46.7	47.0	47.3	47.5
	3	4	2.55	1.9	59.0	41.8	...	...	...	...	...
	4	6	3.84	1.9	59.0	37.3	36.0	36.4	36.7	37.4	37.7
B	5	2	1.29	1.2	55.0	49.5	48.0	48.8	49.9	51.0	...
	6	3	1.84	1.3	57.3	45.6	44.6	45.2	45.5	45.0	44.9
	7	4	2.33	1.6	55.0	40.9	39.7	40.3	40.5	40.9	41.1
	8	6	3.82	2.2	60.6	38.9	37.6	38.1	38.7	38.9	...
C	9	2	1.23	6.2	58.0	49.7	47.9	48.4	49.2	49.9	49.5
	10	3	1.88	6.4	55.7	44.2	43.5	43.9	44.4	44.6	44.4
	11	4	2.41	6.5	58.3	41.8	40.5	41.1	40.9	42.2	42.8
	12	6	3.75	7.1	58.2	38.4	37.7	37.8	38.0	38.0	...
D	17	6	3.96		58.4	37.2	...	...	...	...	...
	18	6	4.18		56.4	35.2	...	...	...	...	...
Averages for sliced specimens						43.3	42.1	42.7	43.1	43.5	44.0

[a] Series A—consolidated-undrained tests with back pressure,
B—consolidated-undrained tests without back pressure,
C—consolidated-undrained tests with back pressure, deviator stress held constant for 5 hr, and
D—consolidated-undrained stage tests with back pressure.

[b] $\bar{\sigma}_c$ = cell pressure—back pressure, kg per sq cm.

[c] $(\bar{\sigma}_1 - \bar{\sigma}_3)_f$ = deviator stress at failure kg per sq cm.

[d] T_f = time to failure, hr.

filter paper drains as described by Bishop and Henkel (**15**) and enclosed in a single thin rubber membrane (0.01 cm thick). In series A, C, and D, cell pressure was then increased until small increments in cell pressure created equal increases in pore water pressure. This always occurred before the cell pressure reached 0.5 kg per sq cm. Cell pressure was then increased to 2.5 kg per sq cm while a back pressure of 0.5 kg per sq cm was maintained on the pore water, using equipment previously described (**16**). Drainage was allowed for one day. For tests at higher cell pressures, daily increments of 1 kg per sq cm were applied, allowing at least two days for drainage under the last increment. In series B, the specimens were consolidated without any back pressure, but just before shearing, the cell pressure and pore water pressure were increased simultaneously by 1 kg per sq cm. After consolidation was complete, the drainage was discontinued, and the specimens were strained by axial loading at a rate of about 2 per cent per hr while pore water pressure was measured at the bottom plate.

In series C, the deviator stress was held constant at about one third of its maximum value for a period of 5 or 6 hr. During this period, the specimens continued to deform, and pore pressures increased slowly. Controlled axial strain was then continued by further axial loading.

The two specimens of series D were subjected to multiple-stage tests. This technique has been described recently by Kenney and Watson (**17**). In these tests, a back pressure was applied to the pore water during consolidation and shear. Consolidation pressures of 2, 3, 4, and 6 kg per sq cm were used in successive stages. After each consolidation stage, the dimensions of the specimen were computed, and an axial load was applied by controlled straining without drainage until a maximum deviator stress was reached.

TEST RESULTS

Test results are summarized in Table II. The relationship between strain and deviator stress and pore water pressure for the four specimens of series A is shown in Fig. 1. Similar results were obtained for the other tests, but the pore pressure measurements for series B (consolidated without back pressure) appeared to be less reliable. For specimen 96-4-7, in particular, the measured pore pressure was low and not consistent with the other tests. In series C, about 0.2 per cent strain occurred during the period of constant deviator stress, and pore pressure increased in proportion to

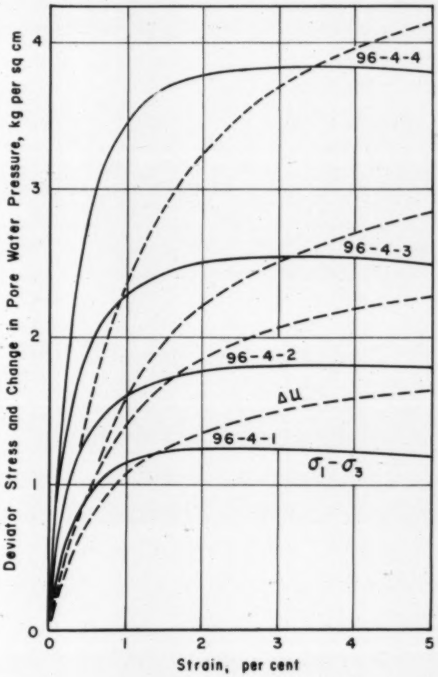

FIG. 1.—Typical Test Results.

strain as shown in Fig. 2. Specimen 96-4-10 was inadvertently stressed to 46 per cent of failure and has therefore greater pore pressures than normal.

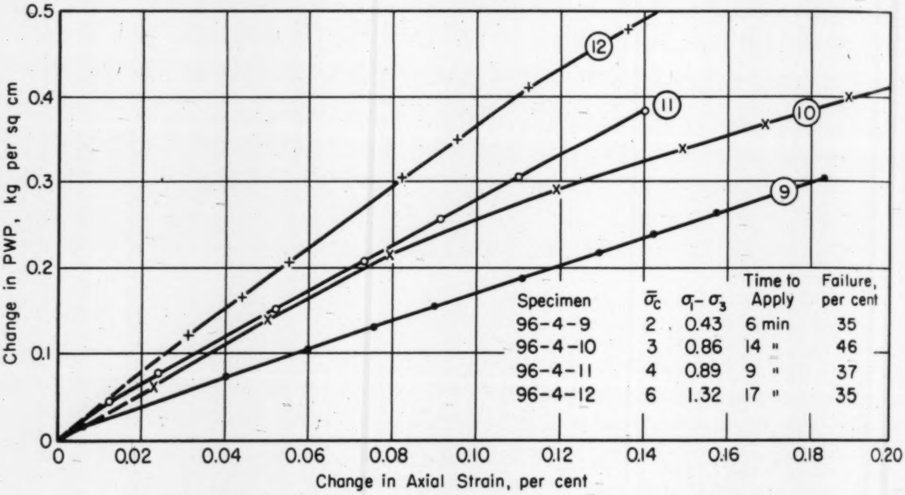

FIG. 2.—Relation Between Pore Pressure and Strain at Constant Stress.

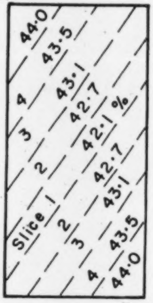

FIG. 3.—Average Water Content of 10 Specimens After Shearing.

Ten of the specimens were removed as quickly as possible from the triaxial chamber and divided into slices parallel to the failure plane in order to measure water content variation between slices. Specimens in which the failure plane was not clearly visible were compressed rapidly in unconfined compression to establish the failure plane. The water content of each slice is given in Table II, and the average variation is shown in Fig. 3. Slice 1 is the slice enclosing the failure plane. The position of the other slices is shown in the figure. The average water content of the ten specimens at failure is 43.3 per cent. The average water content of the slice enclosing the failure plane is 42.1 per cent. The water content of all of the specimens decreased consistently toward the failure plane, averaging 44 per cent at the extremities to 42.1 per cent at the failure plane.

Two specimens from an adjacent block sample were consolidated under one loading increment of 6 kg per sq cm and then removed from the cell quickly without shearing. End slices were removed, and the specimens were divided into upper, middle, and lower sections. Each section was trimmed concentrically into an outer, intermediate, and central portion. Water contents of each portion were determined. These tests revealed a pattern of water content that suggests different degrees of consolidation throughout the specimen. The tests show the outer shell to have been about 1 per cent dryer than the central portion (Fig.

4). Most of slice 1, obtained from sheared specimens, came from the inner portion of the sample, while most of slice 4 or 5, for instance, came from the outer shell, and it is therefore reasoned that slice 1 was wetter than average before shearing. Since it was dryer than average after shearing, this suggests an even greater movement of water during shear than is indicated by measurements on slices parallel to the failure plane.

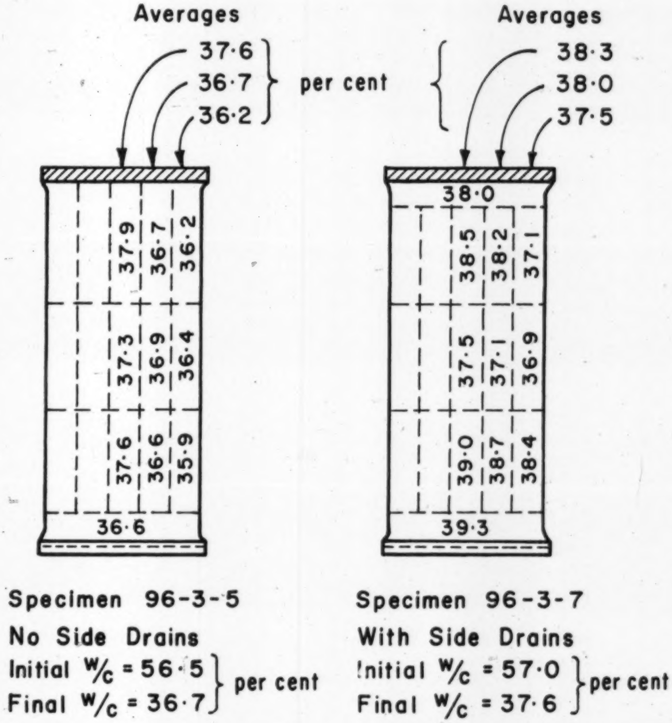
Averages
37·6
36·7
36·2
per cent
Averages
38·3
38·0
37·5
37·9
36·7
36·2
37·3
36·9
36·4
37·6
36·6
35·9
36·6
38·0
38·5
38·2
37·1
37·5
37·1
36·9
39·0
38·7
38·4
39·3
Specimen 96-3-5
No Side Drains
Initial W/c = 56·5
Final W/c = 36·7
per cent
Specimen 96-3-7
With Side Drains
Initial W/c = 57·0
Final W/c = 37·6
per cent

FIG. 4.—Water Content Variation After Triaxial Consolidation.

DISCUSSION OF TEST RESULTS

The fact that the computed angle of shearing resistance, ϕ', of a sensitive clay is subject to a wide interpretation is disturbing. Consider specimen 96-4-4, consolidated under an all-round pressure of 6 kg per sq cm. Since this pressure is well above the natural preconsolidation pressure of the soil, it may be considered to be normally consolidated with $c' = 0$. At 1 per cent strain, when 90 per cent of the maximum deviator stress had been applied, the computed ϕ' was 19 deg, at 95 per cent it was 21 deg, and at 100 per cent it had risen to 26 deg, at a strain of nearly 3 per cent. At approximately 10 per cent strain, the maximum principal effective stress ratio, $\bar{\sigma}_1/\bar{\sigma}_3$, occurred and the computed ϕ' was 34 deg. The application of the last 10 per cent of maximum deviator stress has therefore increased the possible computed ϕ' by as much as 15 deg, but only after considerable strain. This poses the important question of whether or not the triaxial test satisfactorily represents *in situ* conditions of stress and strain for this sensitive, brittle soil.

It is thought that after about 80 to 90 per cent of the maximum stress has been

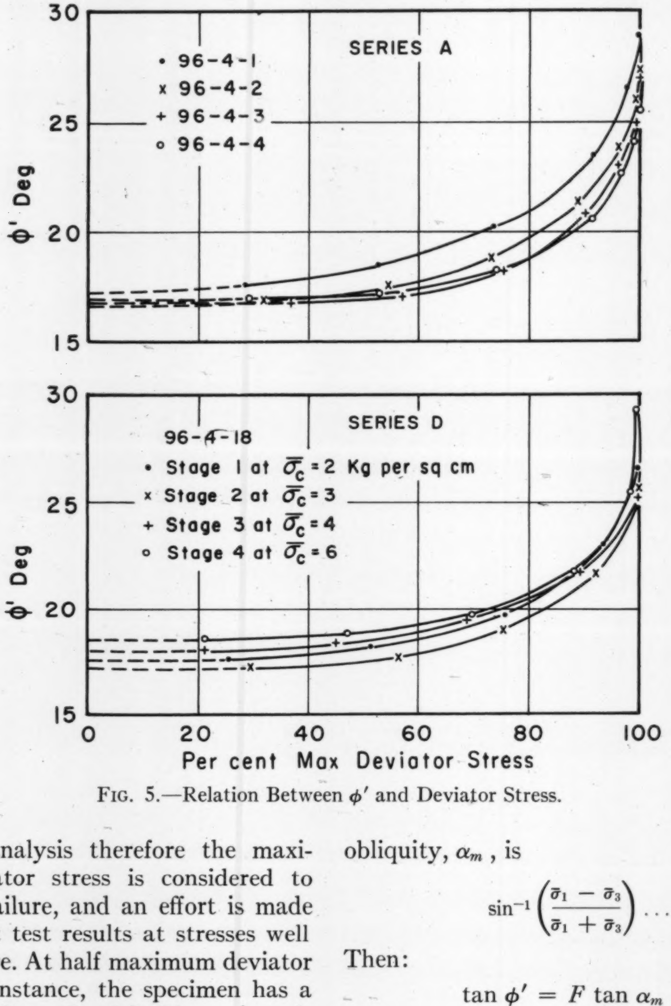

FIG. 5.—Relation Between ϕ' and Deviator Stress.

applied, the soil begins to creep and build up pore pressures which are not compatible with field phenomena. In the following analysis therefore the maximum deviator stress is considered to represent failure, and an effort is made to interpret test results at stresses well below failure. At half maximum deviator stress, for instance, the specimen has a factor of safety, F, of 2. Since $c' = 0$, the shearing resistance at stresses less than failure may be represented by:

$$S = \bar{\sigma} \frac{\tan \phi'}{F} \qquad \text{(1)}$$

where ϕ', the maximum angle of shearing resistance, is equivalent to the "maximum angle of obliquity" at failure as defined by Taylor (18). At half maximum deviator stress, the maximum angle of obliquity, α_m, is

$$\sin^{-1}\left(\frac{\bar{\sigma}_1 - \bar{\sigma}_3}{\bar{\sigma}_1 + \bar{\sigma}_3}\right) \qquad \text{(2)}$$

Then:

$$\tan \phi' = F \tan \alpha_m$$
$$= 2 \tan \alpha_m$$

For specimen 96-4-4, when $F = 2$, $\alpha_m = 9$ deg and $\phi' = 17$ deg.

When ϕ' was computed in this way at various degrees of maximum stress, it was found to be fairly constant up to about half maximum deviator stress. Further

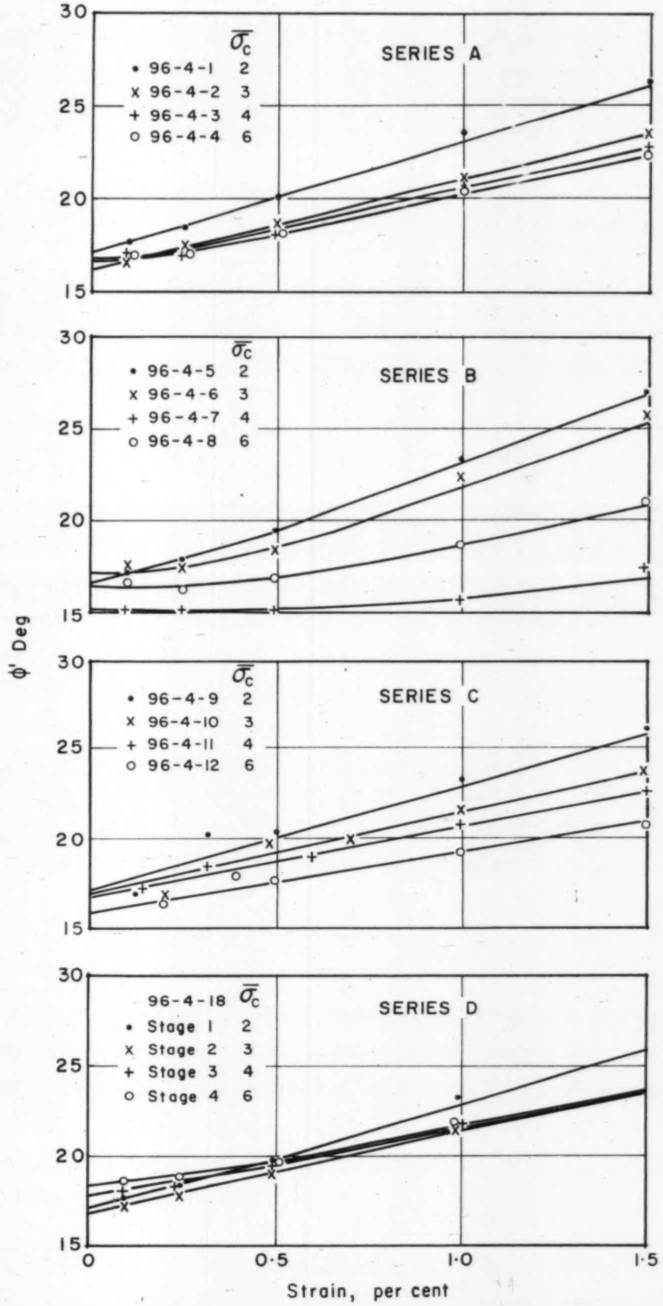

FIG. 6.—Relation Between ϕ' and Percentage Strain (Specimens 96).

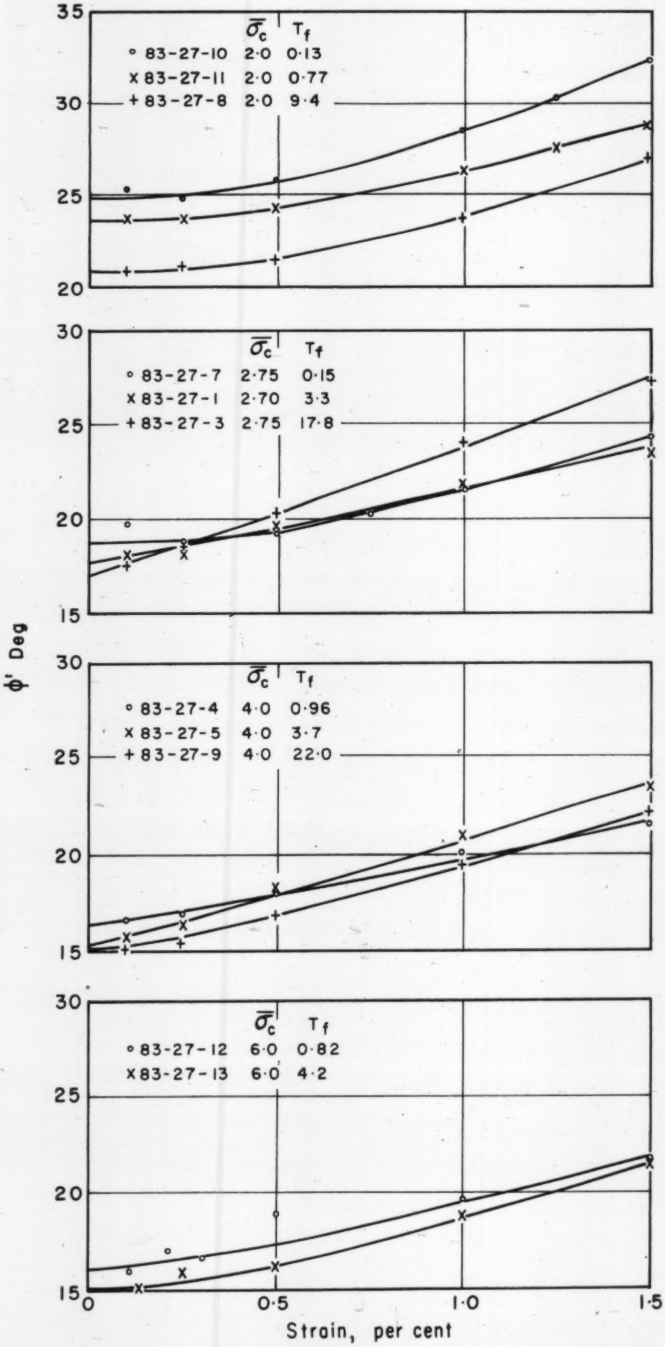

FIG. 7.—Relation Between ϕ' and Percentage Strain (Specimens 83).

stressing caused a marked increase in computed ϕ'. In Fig. 5, values are plotted for specimens of series A and D. A simpler relationship was found between ϕ' and per cent strain. Additional test results plotted in this way are shown in Fig. 6. Curves were drawn through points up to maximum deviator stress, but since the curves were practically straight beyond 1 per cent strain the values are shown only to 1.5 per cent strain.

The significant feature of these relationships is that the computed ϕ' at low stresses is practically constant at an average of about 17 deg and is independent of consolidation pressure. Note that specimen 96-4-1 lies slightly above the average because the laboratory consolidation pressure is slightly less than the *in situ* preconsolidation pressure. Series B test results are scattered, probably because specimens were not consolidated under back pressure and the measured pore pressure is less reliable. Series C shows considerable scatter in the low strain range because ϕ' was computed just before and just after a period of several hours at constant stress. The stage test of series D is especially significant because it agrees with the other tests and can be carried out on only one specimen over a wide stress range.

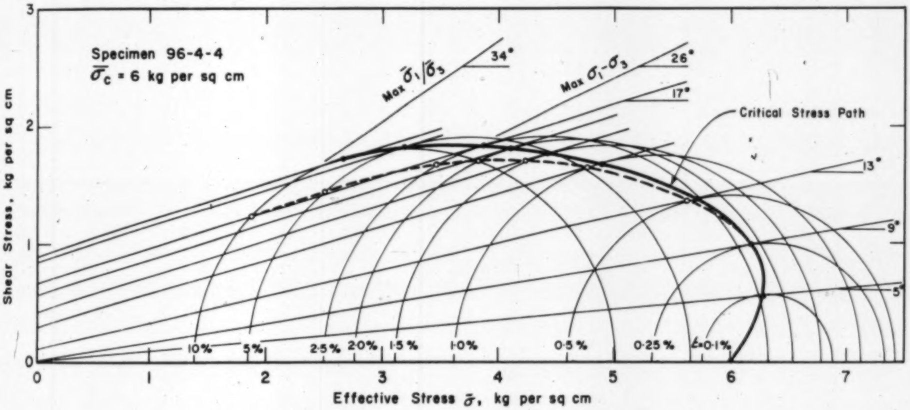

FIG. 8.—Effective Stress Circles at Various Degrees of Strain.

The series of previously published tests on Leda clay (**14**) have been analyzed in the same way and are shown in Fig. 7. These results, which were obtained from samples from another location and in which the rate of strain was varied from about 0.1 per cent to 10 per cent per hour, reveal a similar value for ϕ' of about 15 to 17 deg at low strains which appears to be independent of rate of strain. Tests at consolidation pressures less than 3 kg per sq cm give higher values of ϕ' and appear to be affected by the natural overconsolidation.

From these test results, it is thought that the computed ϕ' of 17 deg is a fundamental parameter equivalent to Hvorslev's true friction angle ϕ_e. For discussion, reference is made to Fig. 8 which shows Mohr stress circles for specimen 96-4-4 at various degrees of strain up to 10 per cent. The maximum angles of obliquity at strains of 0.25 per cent and 0.5 per cent are 5 and 9 deg, respectively, and the computed values of ϕ' are 16.9 and 17.1 deg. In this stress range, rather little change occurs in the effective stress on the plane of maximum obliquity. It is reasonable to suppose that the true frictional resistance is not fully developed. At 2.5 per cent strain, the maximum deviator stress is reached. Since effective stress on the failure plane is reduced to about 60 per cent of its maximum, the frictional resistance is fully developed and the shearing resistance envelope is drawn at an angle of $\phi_e = 17$ deg intersecting the ordinate axis at a value of 0.65 kg per sq cm. This is regarded as the true cohesion, c_e, defined by Hvorslev and is thought to be due to prestressing (19) on the failure plane. Other values at various strains are shown. At 0.5 per cent strain, it is thought that some true cohesion is developed due to prestressing even though friction is not fully developed.

A solid curve is drawn through points of tangency to the Mohr circles to indicate the effective stress on the most critical shear plane which gradually steepens as friction is mobilized. This "critical stress path" is similar to but not quite the same as the "vector curve" employed by Casagrande and Wilson (19). A set of "critical stress paths" obtained in test series A is illustrated in Fig. 9. These indicate that the magnitude of true cohesion is dependent on the consolidation pressure as postulated by Hvorslev for remolded specimens. In the lower part of the figure, the relationship between true cohesion and consolidation pressures is illustrated. The Hvorslev "coefficient of effective cohesion" is 0.11. Although systematic observations have not been made, it has been casually observed that the shear planes at failure in this material are inclined at about 55 deg to the horizontal, giving further confirmation to the reported value of ϕ_e (1).

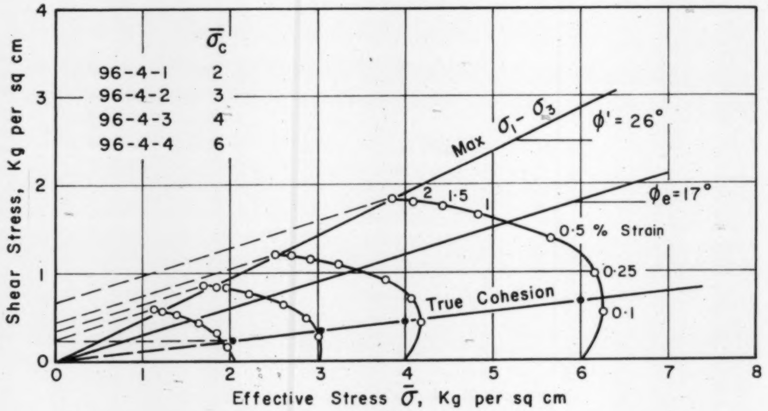

FIG. 9.—Effective Stresses on Most Critical Shear Planes.

DRAINED TESTS

Some consolidated drained tests have been made on this sensitive clay, but none is reported in the paper. It has been found that when axial stress is increased with full drainage, the specimen deforms up to 30 per cent strain or more before maximum strength is reached, and it has been argued that such a test has no practical significance (10,14). If the specimen is caused to fail by decreasing the lateral stress, the relationship between computed ϕ' and strain is similar to that measured by an undrained test, but considerable strain is again necessary for failure (10). The reasoning applied to consolidated undrained tests can not be extended to the drained tests because there is no period of more or less constant effective stress on the failure plane.

For relatively insensitive remolded clay, Henkel (20) found strength in terms of effective stresses to be independent of type of test, drained or undrained, but he was unable "to relate shear strains measured in the various types of tests." He drew attention to the necessity of relating shear stress and shear strain for practical problems and cautioned against extending his laboratory results to field problems.

For both undisturbed and compacted clays, Hirschfeld (21) found the relationship between strength and effective stress to be dependent on test method (drained or undrained). From a study of many test results, he concluded that time of loading and prestress are the most important factors influencing the relation between shear strength and effective stress in a saturated clay.

OTHER SOILS

Test results for a Drammen clay have been reported in sufficient detail by Bjerrum and Simons (9) to permit determination of its "true angle of internal friction" by the described method. This clay, with a liquid limit of 37 per cent, plastic limit of 18 per cent, and natural water content of 34 per cent, is computed to have a true angle of about 18 deg.

Tests described by Simons ((22), Fig. 1) on a highly plastic clay from Canada, for which sufficient information was available, gave a computed true angle of internal friction of about 16 to 17 deg. Tests by the Division of Building Research on samples from the same area gave approximately the same value.

VARIATIONS IN SPECIMEN DURING TEST

The results of several investigations on the stress variation in a specimen during a triaxial test were summarized by Hvorslev (23). Shockley and Ahlvin (24) reported significant stress variation and opposite volume change tendencies in various portions of sand specimens being sheared in a triaxial device and concluded that "interpretations of test data based on total volume changes may be completely at variance with those determined on the basis of changes in the failure zone alone." The late D. W. Taylor reported that "non-uniform conditions of stress and strain occur in all types of tests, and failures are always, to a degree, progressive" (18). Taylor measured pore pressures within clay specimens, and after shear he measured the variation in water content. His results, just recently published by Whitman (25), show variations of $1\frac{1}{2}$ per cent. Similar results are quoted in this paper.

It is difficult, if not impossible, to make allowance for stress variation and water migration within a specimen during the triaxial test. It is useful, however, to attempt to judge the significance of these complications in special tests on reasonably uniform specimens. This is done with reference to Fig. 8. Initially, it is reasoned that as the shear stress approaches its maximum value the structure of the sensitive clay breaks down and transfers normal stresses to the pore water, causing water to flow from the most critical plane. As friction develops progressively, the critical plane steepens to an angle of $45 + \frac{\phi_e}{2}$ to the horizontal. If the material remained homogeneous, the critical plane would maintain its position, but if consolidation occurs on the plane, it will tend to move up or down or to steepen. If a progressively steepening plane due to mobilization of friction is visualized, it is thought that this trend will continue because a shift up or down in the specimen would require the critical plane to move into a region previously affected by consolidation. If the critical plane steepens due to progressive drying, the failure envelope will intersect the Mohr circles which are computed from total stresses minus measured pore water pressure, and the "critical stress path" will trend toward that shown by a broken curve in Fig. 8. Casagrande and Wilson (**19**) used similar reasoning to explain variation in the shape of "vector curves" depending on type and rate of stressing.

FRICTION AND COHESION

There has not been satisfactory agreement on the true mechanism of friction and cohesion in clay soils. On the basis of experiments with remolded clays, Rowe (**26**) suggested that true cohesion may be unstressed at equilibrium and that creep would result if true cohesion were mobilized. Lambe (**27**) saw no fundamental difference between the mechanism of friction and cohesion. He argued (**28**) that cohesion was mobilized at very small strains, and at some further degree of strain it ceased to contribute to shearing resistance. From a comprehensive set of tests on a variety of soils, Schmertmann and Osterberg (**29**) conclude that the "cohesion strength component generally develops to its maximum value at an axial compressive strain of less than 1 per cent, while the friction angle requires a much greater strain to reach its maximum value." For normally consolidated Lilla Edet clay, Bjerrum and Wu (**7**) found true cohesion to be proportional to the consolidation pressure. Specimens reconsolidated in the laboratory to pressures less than the preconsolidation pressure were found to have additional true cohesion. This they attributed to rigid bonds between particles that suddenly break down at pressures above the natural preconsolidation pressure.

In a well-documented paper, Rosenqvist (**30**) traced the development of physicochemical concepts of soil water systems and emphasized the importance of considering a portion of the water in a soil as part of the soil particles. This concept led him to conclude that when the pressure between mineral grains is released they will continue to stick together because of adhesional forces. This is thought to be a most important concept and is employed in the following explanation for the development of friction and cohesion in the Leda clay.

It is believed, based on test results reported in this paper, that all shearing resistance is of a frictional nature (that is, dependent on normal effective stress on the plane of shear) and that cohesion is caused by the inability of the interparticle forces to decrease quickly (Rosenqvist's adhesion) when external loads are released. It follows that the frictional component (ϕ_e = 17 deg) is independent of time or stress path. The cohesion component, on the other hand, is dependent on stress release and may be expected to begin to dissipate as soon as it is mobilized. It will probably not decrease to zero. Soils which have been rebounded geologically might be expected to retain a minimum cohesion. This could account for the "threshold effect" observed by Bjerrum and Wu (**7**).

Friction in clays will probably not be as simple as friction between ordinary plane surfaces since it is not thought to involve direct mineral contact. Rather, it will be increased by decreasing distance between particles and by enlarged overlapping of water films. It is probable that the decreasing distance between mineral grains brought about by increasing stress is not compatible with the minerals' demand for complete surface layers of water and that long term adjustment will take place as particles move relative to one another seeking equilibrium. This adjustment is considered to reduce the overlapping of water films with a consequent reduction in the so-called "true cohesion." Time-dependent decrease in the empirical cohesion intercept c' has been shown in the laboratory (**14**) and in the field (**31**), and this may logically be interpreted to indicate similar variation in the true cohesion c_e.

APPLICATION

The Hvorslev parameters are considered to represent the correct mechanism of shearing resistance in a soil. Empirical parameters in terms of effective stresses are thought to provide a satisfactory basis for stability analyses provided the stress range during test corresponds to that in the field. A valuable comprehensive study of evidence supporting this view has been presented recently by Bishop and Bjerrum (**32**). The correlations between analysis and performance always appear to be less satisfactory for natural soils than for compacted soils. This is usually attributed to greater variations in the properties of natural soils and to unknown influences of weathering. A further, and perhaps more important, reason may be errors in the laboratory values of temporary and permanent cohesion, especially in the low stress range. It is not easy to evaluate the true strength parameters in the over-consolidated range, but one of the most difficult soil engineering problems involves unloading of natural soils. It is clear therefore that much more work must be done on natural soils in this important stress range.

CONCLUSIONS

The following conclusions are based on relatively few consolidated undrained test results on a sensitive marine clay. Some evidence of a certain general applicability is quoted, but the extrapolation of test results on sensitive clays is thought to be hazardous.

1. The measurement of shear strength parameters of a sensitive clay in terms of effective stresses applied in the triaxial compression apparatus is subject to an unsatisfactory range of interpretation.
2. The computed ϕ' is directly related to strain in the specimen. At stresses up to about one half of failure stress in the material tested, it is reasonably constant at about 17 deg. On the basis of the Hvorslev hypothesis, this angle is thought to be equivalent to the "true angle of internal friction."
3. This "true angle of internal friction" is independent of cell pressure and rate of strain.
4. "True cohesion" is considered to be equivalent to a frictional phenomenon caused by intrinsic pressures which are not immediately reversible when stresses are released but which will decrease with time.
5. True friction is considered to be mobilized at very low strain (less than 1 per cent in the specimen considered) with true cohesion mobilized as required during stress release.
6. Measurements show that nonuniform consolidation occurs due to application of all-round pressure and that water moves away from the plane of shear. This is thought to influence effective stress parameters, especially at high strains.
7. Most reliable pore pressure measurements are obtained when specimens are consolidated under a back pressure.
8. Under constant axial stress, pore pressure increases in direct proportion to strain.

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LIST OF SYMBOLS

σ = normal stress, kg per sq cm,
u = pore water pressure, kg per sq cm,
Δu = pore pressure change during shear, kg per sq cm,
$\bar{\sigma}$ = effective stress, kg per sq cm,
$\bar{\sigma}_c$ = effective consolidation stress, kg per sq cm,
$(\bar{\sigma}_1 - \bar{\sigma}_3)_f$ = deviator stress at failure, kg per sq cm,
T_f = time to failure, hr,
F = factor of safety,
α_m = maximum angle of obliquity,
c' = cohesion intercept in terms of effective stresses, kg per sq cm,
ϕ' = angle of shearing resistance in terms of effective stresses,
c_e = true cohesion, kg per sq cm, and
ϕ_e = true angle of internal friction.

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DISCUSSION

MR. WERNER E. SCHMID[1] (*presented in written form*).—Mr. Crawford presents a series of most interesting results and he should be commended for the diligence with which the reported experiments were carried out. By far the most significant results of his investigations are the wide variations that are possible in the interpretation and evaluation of shear strength data with pore pressure measurements. The effective angle of shearing resistance ϕ' is found to vary, depending on the strain, from $\phi' = 19$ deg to $\phi' = 34$ deg. Similar results have been observed also by this writer. Such wide variations must clearly be disturbing to anyone who has to use laboratory data of such nature for the design of field installations. These variations, of course, result from the build up of pore pressure with strain and time. For this and a number of other reasons, we have concluded it is unwise to assess the shear strength of a clay soil in the field from laboratory triaxial tests with pore pressure measurements. They are subject to too wide a fluctuation depending entirely upon the strain at which the tests are evaluated, the compliance of the measuring system, the point of pressure pickup, etc.

As a consequence, we are proposing an entirely different shear strength theory[2, 3] which redefines the whole problem and introduces a new concept of shearing strength. A detailed exposition of these concepts is beyond the scope of this discussion; however, the basic principles are outlined briefly:

Clay Soil as an Engineering Material.—It is generally known and accepted that the composition of an engineering material is the most important factor that determines its properties. We utilize this knowledge to manipulate the properties of alloys and of plastics, as well as other materials. The carbon content of a steel determines its yield strength and ductility, and the water-cement ratio of a concrete is the most critical parameter for concrete strength. The composition parameters are very sensitive; that is, their slightest change has a great effect upon the material properties. When we conduct triaxial tests in the soil mechanics laboratory, the samples are usually consolidated at different lateral pressures. This results in different water contents. Hence, samples thus consolidated ***are really different engineering materials!*** In view of our experience of the sensitivity of composition parameters, the notion that the change in shear strength of a whole series of engineering materials can be expressed by one constant parameter ϕ' in the equation $s = c + \sigma \tan \phi'$ appears to be a vain and naive hope.

In order to get significant and meaningful results we should, therefore, carry out our tests on samples that have the same composition, that is, the same water content. If such triaxial tests are carried out (undrained quick tests), we find that on the basis of total stresses the failure envelope in a Mohr diagram is a horizontal line, that is, $\phi = 0$. This means that the Mohr-Coulomb failure theory is not applicable, the shear strength *does not* directly depend on the normal stresses, and the general acceptance of the Mohr-Coulomb theory stems from a confusion of cause and effect.

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[2] W. E. Schmid, Y. Klausner, and C. F. Whitmore, "Rheological Shear and Consolidation Behavior of Clay Soils," Progress Report to the Office of Naval Research, Princeton, University, (1960).

[3] W. E. Schmid, "New Concepts of Shear Strength for Clay Soils," *Journal*, Soil Mechanics Div., Am. Soc. Civil Engrs. (in press).

Shear Strength of Saturated Clay Soils.—On the basis of the concepts above, we formulated a shear strength concept which does not separate s into friction and cohesion components but considers the total value of the shear strength which is found to depend on (*a*) the water content of the clay soil and (*b*) the duration of the load. It is also proposed that the best measure for shear strength is the octahedral shear stress. Since the maximum shear stress, $\frac{1}{2}(\sigma_1 - \sigma_3)$, varies only by—at most—15 per cent from the octahedral shear,[4] the former may be substituted without significant loss of accuracy. Thus we may write:

$$s = \frac{1}{2}(\sigma_1 - \sigma_3)_{\max} = Ae^{B(w-w_0)}$$

where:

A and B are material constants that vary with the load duration, and

e is the base of the Naperian logarithm.

Water Content Variation.—In view of the concepts above, the variations of the water content within a triaxial specimen become most interesting, and Crawford's data are highly significant. They confirm observations in our laboratory where within a research program on shear strengths of clay soils we observed the variation of water content within triaxial test specimens before and after shearing. Our specimens were made of remolded, homogeneous, saturated clay of illite and—in the second series—kaolinite type clay minerals. We found that:

1. After consolidation (with side drains) the water contents were highest at the bottom of the specimens, somewhat lower at the top, and lowest at mid-height. Radially, the water content decreased from a maximum at the center to a minimum at the edge. The range of the differences was between 1 and 2 per cent.
2. After shearing, the failure zone always had the smallest water content with deviations from the average water content of the sample by again 1 to 2 per cent.

Deviator Stress versus Stress Difference.—The only displeasure this writer has with Mr. Crawford's otherwise excellent paper is the unfortunate choice of the term stress deviator for the principal stress difference $\sigma_1 - \sigma_3$. While we recognize that this mistake is made frequently in soil mechanics circles, we feel it is time to correct this situation and avoid this constant source of confusion. The term deviator stress has been defined in classical mechanics as the difference between the principal stress and the mean hydrostatic stress, and we should respect this definition:

$$\text{Deviator stress } S_1 = \sigma_1 - \frac{1}{3}(\sigma_1 + \sigma_2 + \sigma_3).$$

Inclination of Failure Surface.—Saada[5] has shown recently that the inclination of the failure surface on a triaxial specimen should be equal to the octahedral angle $\alpha = 54°45'$. This is in excellent agreement with the inclination of 55 deg observed by Crawford as well as Saada's own observations.

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[5] A. S. Saada, "Rheological Investigation into the Shear Behavior of Saturated Clay Soils," Ph.D. Dissertation, Princeton University (1961).

MR. DONALD M. BURMISTER[6] (*presented in written form*).—The influences of strain on stress-strain and shearing strength responses of clays appears to be more characteristic of certain preconditioned states than of inherent sensitivity of clay as such. Soil is a most unusual construction material because its responses in laboratory tests and its field performances with regard to foundation support of structures and stability of earthworks and natural slopes are strongly influenced and conditioned by the kind, relative dominance, and sequences of conditioned imposed. There are no simple model-prototype relations between laboratory soil responses and field performances which can be used invariably and reliably. Soil is always a prestressed and prestrained material with a prestress at any depth at least equal to the weight of overburden.

A first fundamental concept of soil performances states that soils having the same identifications but different prestress, confinement, and preconditioning are in reality different materials, because they possess essentially different responses and performances. A sample of clay which has been taken from its prestress and confinement environment is a radically different material from that in the natural "in place" state. Furthermore, soil samples may be adversely altered in some degree by sampling disturbances. A second fundamental concept, therefore, follows in accordance with the principles of prototype triaxial testing of soils that the original prestress and confinement conditions must be restored on a triaxial specimen, and thereafter the new conditions imposed by construction of structures and earthworks must be applied, all with proper regard to kind, relative dominance, and sequences of conditions. Thus prototype stress-strain and shearing strength responses of clay soils can be consistently and reliably obtained that have direct and valid applications in evaluation and solutions of foundation and stability problems and in predicting full scale field performances.

The prestressing of triaxial specimens of clay by complete consolidation to increasing stress levels corresponding to different depths represents only one aspect of prototype testing. The prior determination of the natural prestress, state of consolidation, and undisturbed quality and "goodness" of samples are essential in triaxial testing in order to set up proper triaxial test conditions and procedures. Prototype consolidation tests are required with an unloading-reloading stress cycle and characteristic triangle construction to establish the prestress and state of consolidation.[7] If a clay is normally consolidated with the prestress equal to the present overburden, the starting point of straining "in place" under imposed stresses is at this prestress. The significant and usable stress region for triaxial testing is therefore just above this natural prestress level on the primary consolidation curve and associated triaxial curve. Stress-strain responses below the prestress are merely a laboratory reloading phenomenon to restore this prestress.[8]

More frequently than is supposed, clays have been over-consolidated under prestresses greater than the present overburden. For over-consolidated clays, the starting point of straining "in place" under imposed stresses is at the overburden stress in a natural unloading stress cycle. The significant and usable stress region for triaxial testing is on a relatively flat reloading stress cycle just above the overburden stress to the natural prestress and continuing thereafter on a steeper primary consolidation curve and associated triaxial curve.[8]

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[6] Civil Engineering Dept., Columbia University, New York, N. Y.

[7] D. M. Burmister, "Strain-Rate Behavior of Clay and Organic Solids," Papers on Soils—1959 Meetings, *ASTM STP No. 254*, Am. Soc. Testing Mats., pp. 90–92, Fig. 1, pp. 99–104 (1959).

[8] D. M. Burmister, "The Importance of Natural Controlling Conditions upon Triaxial Compression Test Conditions," Triaxial Testing of Soils and Bituminous Mixtures, *ASTM STP No. 106*, Am. Soc. Testing Mats., pp. 256–261, Figs. 1 and 2 (1950).

Furthermore, the loading and confinement conditions in sequence have important influences on stress-strain responses and mobilizable shearing strength for foundation support, but more critically for stability of structures and earthworks under unbalanced stress conditions. The strains in the latter case are much smaller—first, for unloading conditions, such as exposure of a slope by excavation and resulting instability conditions and, second, for conditions of lateral yielding of retaining structures with accompanying relief of stress from the "earth pressure at rest" to the active pressure state.[9] Prototype triaxial testing requires that these strain and lateral confinement conditions be simulated by proper testing procedures in sequence which will have to be learned by experience. In addition, the initial prestressing and preconsolidation of triaxial specimens to required stress levels should be in accordance with vertical and lateral stresses increased in a definite ratio corresponding to the state of consolidation, instead of by hydrostatic preconsolidation.[9]

The controlling influences of slow plastic creep, however, are considered to be more important than the above influences for clay soils. Creep phenomena were treated rather completely in the paper entitled—"Strain-Rate Behavior of Clay and Organic Soils."[7] In the period from 1933 to date, there have been many opportunities to observe creep phenomena in action, to study the nature and controlling influences of creep, to evaluate the conditions that govern creep, and to design bulkheads, retaining structures, earth dams, and embankments over clay and organic soil deposits against failure eventually by slow creep. Methods and apparatus for triaxial creep testing were developed and used since 1945. Cases 1 and 2 describe the nature of the problems involved in stability of embankments.[7]

Observations, experiences, and investigations of mass slide failures have shown that a mass slide has practically always been initiated by a phase of slow plastic creep.[7] The initial elastic slumping downward of a slope induces shearing stresses beneath the toe of a slope. If these shearing stresses fall within the range of appreciable creep strain-rates, then creep is initiated beneath the toe of the slope, which is the stress region most vulnerable to creep action. When lateral subsurface creep displacements accumulate to sufficient magnitudes beneath the toe of a slope, settlement of this toe portion must inevitably result. This, in a real sense, "pulls the rug" from under the slope and tends to tilt the slope or embankment outward. As a consequence, well-known tension cracks are formed back of the crest of the slope, which are evidence of creep conditions. Some shearing strength necessary for stability is thus destroyed progressively as these cracks deepen, causing a greater unbalance of stress conditions. As creep is now propagated back beneath the slope, the creep displacements accumulate finally to critical magnitudes. Eventually, these sequences of events lead to a rapid mass slide failure, especially if heavy rains fill the tension cracks and induce additional sliding forces, as a "trigger" action.

Creep is essentially a stress-deformation stress-relaxation phenomenon,

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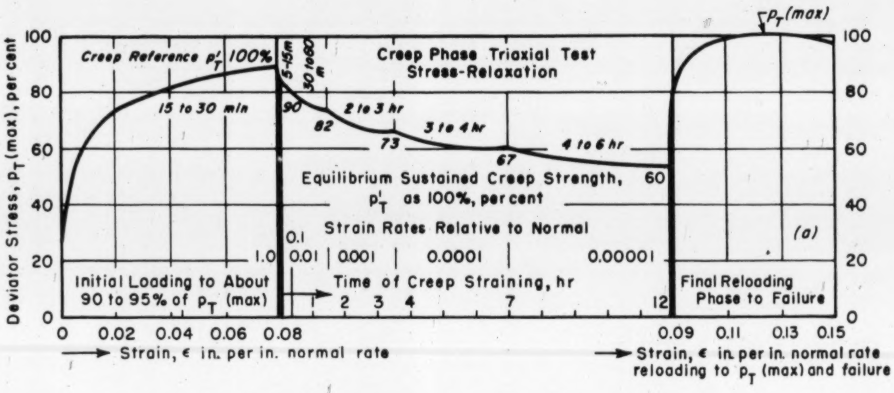
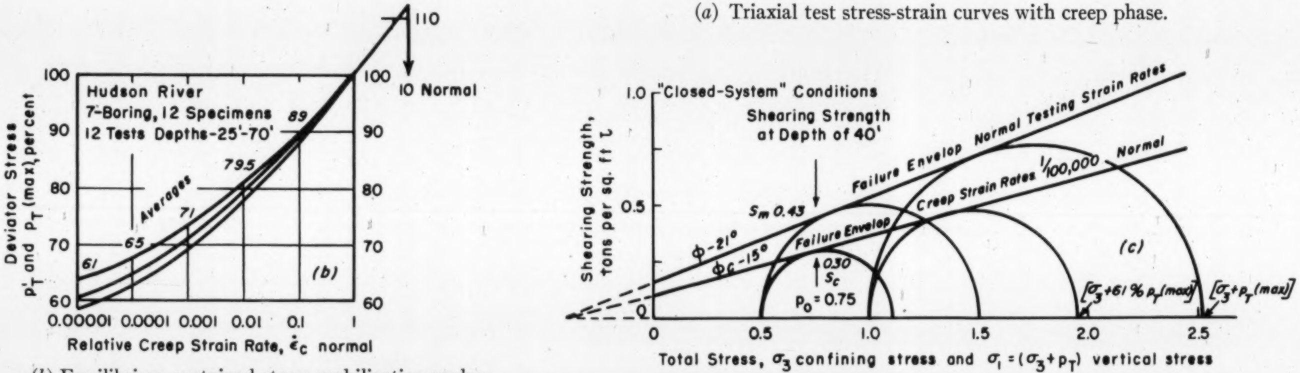
[9] D. M. Burmister, "The Place of the Direct Shear Test in Soil Machines," Symposium on Direct Shear Testing of Soils, *ASTM STP No. 131*, Am. Soc. Testing Mats., pp. 6–11, Figs. 3 and 4 (1952).



(*a*) Triaxial test stress-strain curves with creep phase.



(*b*) Equilibrium sustained stress mobilization under decreasing creep strain rates. A stress relaxation phenomenon.

(*c*) Mohr stress circle analysis of failure conditions at normal triaxial testing rates and creep strain rates 1/100,000th normal.

FIG. 10.—Prototype Triaxial Stress-Strain and Creep Responses to Determine Equilibrium Sustained Creep Shearing Strength. (Dark gray ORGANIC SILT, little fine sand, low plasticity range and average moisture content.)

which is illustrated in Fig. 10(*a*). A preconsolidated triaxial specimen is strained under "closed system" conditions by application of a triaxial or deviator stress, P_T, to about 90 per cent of the maximum mobilizable stress. Thereafter, a creep phase of testing is performed in five stages with the creep strain rate being reduced successively to $\frac{1}{10}$ of the previous strain-rate and finally to a value of $\frac{1}{100{,}000}$ of normal triaxial testing strain-rates. The creep strain-rate was held constant at each stage until the deviator stress under strain control loading backed off by stress-relaxation to a stable equilibrium sustained stress value for that strain-rate. The length of time to reach equilibrium conditions, as noted in Fig. 10(*a*), increased considerably at the lower strain-rates. The creep strain-rate of $\frac{1}{100{,}000}$ normal is of the order of the very slow creep rates which may be expected to occur in natural clay deposits.

There are a number of important facts concerning creep phenomena which should be recognized and understood. First, the equilibrium sustained creep strength is strain-rate dependent. Second, an equilibrium sustained creep stress is possible at each strain-rate level with creep continuing indefinitely at that rate to final failure. Third, the sustained creep stress in Fig. 10(*b*) tends by the curved relationship toward a rather definite minimum yield value. Fourth, in a Mohr stress circle analysis in Fig. 10(*c*) for total stresses under "closed system" conditions, the creep failure envelope for $\frac{1}{100{,}000}$ normal strain rates falls considerably below the maximum failure envelope for normal testing strain rates. Fifth, this creep phenomenon cannot be attributed to a serious breakdown of soil structure by shearing disturbance effects at this shearing displacement but represents a stress-relaxation characteristic of creep in clay and organic soils. Figure 10(*a*) shows in a rerun test phase at normal testing strain rates immediately after the creep phase that the maximum shearing strength is still fully mobilizable. Sixth, it is evident that cohesion and angle of friction are not stable inherent properties of clay and organic soils, as supposed, but are subject to stress-relaxation influences. Furthermore, cohesion cannot be separated strictly from friction because cohesion is really only a "memory" of a previous prestressed friction state. Seventh, it becomes evident that shearing failure occurs under creep conditions when the accumulated creep displacements exceed a value where a "slip plane pattern" is finally fully developed with an accompanying permanent loss of some shearing strength. By ignoring creep conditions, a factor of safety of 1.4 using an indicated shearing strength of 0.43 tons per sq ft on the maximum failure envelope of Fig. 10(*c*) would be completely wiped out under creep conditions with a mobilizable sustained creep shearing strength of 0.30 tons per sq ft, and failure would be inevitable when the creep strains have accumulated to such magnitude as to develop the failure slip plane pattern with the sustained creep shearing stress never rising above the creep level.

In view of these considerations, a fundamental concept may be stated that if the shearing stresses at the toe of a slope or embankment can be kept with a reasonable factor of safety of 1.25 to 1.50 below a value where creep cannot proceed at strain rates greater than $\frac{1}{100{,}000}$ normal, then creep shearing displacements would not accumulate to sufficient magnitudes within the lifetime of a structure to initiate the final stages of failure by a mass slide, provided that no new or unaccounted for conditions developed not covered by the above margin of safety.

MR. JOHN H. SCHMERTMANN[10] (*by*

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[10] Assistant Professor, Civil Engineering Dept., University of Florida, Gainesville, Fla.

letter).—The many contributions of this very interesting paper by Mr. Crawford include a possible new method for the computation of Hvorslev's effective, or "true," friction component of soil strength, ϕ_e. This method offers the tremendous advantage of obtaining ϕ_e from a single undrained test with pore-pressure measurements. *If valid*, the present difficulty of obtaining the necessary group of identical test specimens would be overcome, and the testing of natural soils for ϕ_e and c_e would be practical. The Hvorslev components would then be freed for use in the practice of soil engineering. In view of its potential importance, this discusser herein further examines the validity of the proposed method of determining ϕ_e.

Conforming to the author's definitions and notation, the following is a derivation of another expression for tan ϕ' to be used in subsequent discussion:

$$\tan \phi' = F \tan \alpha_m = F \tan \left[\sin^{-1} \frac{\bar{\sigma}_1 - \bar{\sigma}_3}{\bar{\sigma}_1 + \bar{\sigma}_3} \right]$$

$$\text{or:} \quad \tan \phi' = \frac{F}{2} \frac{\bar{\sigma}_1 - \bar{\sigma}_3}{\sqrt{\bar{\sigma}_1 \bar{\sigma}_3}} \ldots\ldots (1)$$

In an axial compression, triaxial test

$$\bar{\sigma}_1 = \bar{\sigma}_3 + \frac{(\bar{\sigma}_1 - \bar{\sigma}_3)_f}{F} \ldots\ldots (2)$$

Substitution of Eq 2 into Eq 1 gives

$$\tan \phi' = \frac{1}{2} \frac{(\bar{\sigma}_1 - \bar{\sigma}_3)_f}{\sqrt{\bar{\sigma}_3^2 + \dfrac{\bar{\sigma}_3(\bar{\sigma}_1 - \bar{\sigma}_3)_f}{F}}} \ldots (3)$$

Since $F > 2$ and $\bar{\sigma}_3 > (\bar{\sigma}_1 - \bar{\sigma}_3)_f$ during the deviator stress range considered, this permits $\sqrt{\bar{\sigma}_3^2 + \dfrac{\bar{\sigma}_3(\bar{\sigma}_1 - \bar{\sigma}_3)_f}{F}}$ to be approximated by $\bar{\sigma}_3$ and Eq 3 is simplified to

$$\boxed{\tan \phi' = \frac{1}{2} \frac{(\bar{\sigma}_1 - \bar{\sigma}_3)_f}{\bar{\sigma}_3}} \ldots\ldots (4)$$

Applying Eq 4 to two of the typical tests detailed in Fig. 1, and using the data in Table II with the assumption that $\bar{\sigma}_3 = \bar{\sigma}_c$, which is equivalent to assuming negligible Δu developed during compression, the following comparative results are obtained:

Test No.	$\bar{\sigma}_c$, kg per sq cm	Values of ϕ', deg		
		From Eq 4	From Fig. 5	
			0 per cent Deviator Stress	50 per cent Maximum Deviator Stress
96-4-2 ..	3	16.8	16.6	17.3
96-4-4 ..	6	17.7	17.0	17.1

This agreement indicates that Eq 4 is a good approximation, and also that negligible pore pressure developed during the 0 to 50 per cent range of maximum deviator stress.

A major portion of the author's argument to support his conclusion that the low-strain ϕ' and the Hvorslev ϕ_e are equal is that the computed values of ϕ' are approximately constant from 0 to 50 per cent maximum deviator stress and are approximately independent of consolidation pressure in the normal consolidation range. From Eq 4 it is obvious that ϕ' is approximately constant if $\bar{\sigma}_3$ is approximately constant. This occurred in the author's tests because only small pore pressure developed during strain to 50 per cent maximum deviator stress. It may well occur with most cohesive soils. However, attaching a special true-friction significance to this pore-pressure behavior may be unjustified. First, we must learn the reason for this behavior and then, if possible, relate it to a carefully defined friction or cohesion component. This has not been done.

Replacing $\bar{\sigma}_3$ with $\bar{\sigma}_c$ in Eq 4, which is a reasonable approximation when Δu is small, makes tan ϕ' equal to the well-known c/p ratio. This ratio is often shown graphically, on a semi-log plot with void ratio, as a parallel relationship between normal consolidating pressure and strength. It thus conveniently illustrates that the strength of normally consolidated clay is some constant proportion of the consolidating pressure. Since the clay also decreases in void ratio with increasing pressure, it presumably increases its true cohesion capability. Why then, if $\tan \phi' = \tan \bar{\sigma}_e$, should true friction be the only variable determining this proportion?

An additional argument against considering $\phi' = \phi_e$ is their inconsistency at zero strain. At this strain the shear strength mobilized to resist the external hydrostatic consolidation pressure is zero. The Mohr stress circle is a point on the $\bar{\sigma}$ axis. The sum of the true cohesion, c_e, and the true friction, $\bar{\sigma} \tan \phi_e$, components of soil strength mobilized at zero strain is then also zero. Since $\bar{\sigma}$ is equal to $\bar{\sigma}_c$, either $c_e = -\bar{\sigma}_c \tan \phi_e$ or both c_e and ϕ_e are zero. Rejecting negative values for cohesion or friction angle the conclusion is that ϕ_e must equal zero at zero strain. Again referring to Eq 4, which is exact at zero strain because F is infinite, we see that the defined ϕ' cannot be zero at zero strain. In Figs. 6 and 7, the author shows ϕ' to have values from 15 to 18 deg at zero strain, but ϕ_e must be zero.

This discusser considers that the author's conclusions 2 and 5 are not substantiated. Consequently, the validity of the suggested method for determining the true strength components from a single undrained test is in doubt.

As stated by the author in his quotation from Schmertmann and Osterberg (29), this discusser believes that the low-strain strength of a cohesive soil is primarily due to cohesion. In this reference, cohesion is defined as that component of mobilized shear strength, at any strain and with constant structure, which appears independent of changes in effective stress. There is a great deal of evidence, both published[11] and unpublished, from the results of CFS-tests[12] to support this opinion.

MR. C. B. CRAWFORD (*author's closure*).—Mr. Schmid emphasizes the fact that in a series of consolidated undrained triaxial compression tests, each specimen is at a different water content and is therefore a different engineering material. In the author's view this statement is valid only because the volume deformation of a laboratory specimen under changing effective stress is probably much different than that of an element of soil *in situ*. Since effective stress parameters c' and ϕ' are not fundamental cohesion and friction components, it is clear that the more they depart from the equivalent fundamental components, the greater is the danger of misunderstanding.

The argument that the Mohr-Coulomb failure theory does not apply for undrained tests on saturated clay at constant water content is not valid. According to the "principle of effective stresses," shearing resistance depends on the effective stress on the failure plane, and since in an undrained test at constant water content there is no change in effective stress (no matter what total stress is applied), there is correspondingly no change in shear strength. This concept has been explained by Rutledge,[13] and the limitations of the $\phi = 0$ analysis have been described by Skempton.[14]

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[11] J. H. Schmertmann and J. R. Hall, Jr., "Cohesion After Non-Hydrostatic Consolidation," *Journal*, Soil Mechanics and Foundations Division, Am. Soc. Civil Engrs., August, 1961, pp. 39–60.

[12] The CFS-test is a triaxial test especially devised to permit the determination of cohesion and friction as functions of strain. Details are presented in the references cited.

[13] P. C. Rutledge, "Review of the Cooperative Triaxial Shear Research Program of the Corps of Engineers," Waterways Experiment Station, Vicksburg, Miss. (1947).

Mr. Schmid's observations of water content variation in a specimen due to shear confirm those reported in the paper. This water movement will also be a test difficulty even though shear strength is not divided into cohesion and friction components. The objection to the term "deviator stress" is valid even though usage is well established in soil mechanics literature.[15]

Mr. Burmister draws attention to the important considerations of environment between test and prototype. This cannot be overemphasized. It may be the source of most of our difficulties. In particular, he points out the need in testing natural soils to reestablish the original prestress and confinement conditions that existed before sampling and then to observe the stress-strain response in the appropriate working stress region. It may be pointed out that since deformation depends only on changes in effective stress, the working stress region should be in terms of effective stress.

Mr. Burmister goes on to explain features of creep phenomena based on observations in terms of total stresses on a "closed system" or undrained test condition. His fifth point that "creep phenomena cannot be attributed to a serious breakdown of soil structure" is at variance with ideas expressed in the paper. The reason for this appears to be due to the difference in interpretation depending on whether total stress of effective stress information is available.

Triaxial tests on Leda clay (unpublished) indicate that specimens can be subjected to unloading cycles or to creep under constant load without significantly changing the ultimate shearing resistance in terms of total stress provided that times to failure are not significantly different. This confirms Mr. Burmister's observations in Fig. 10(a) and leads to his conclusion regarding structural breakdown. If pore pressures are measured, however, it can be demonstrated that the development of pore pressure is directly related to strain (Fig. 2). The only explanation for this seems to be that the sensitive structure breaks down during shear strain causing local con-

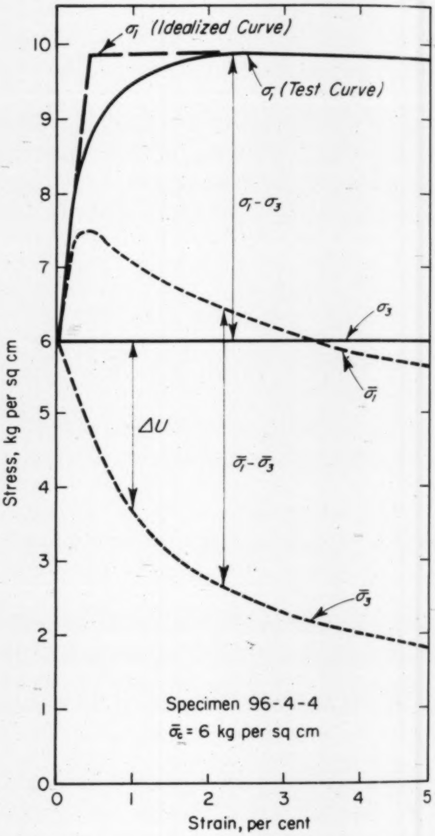

FIG. 11.—Total and Effective Principal Stress Variation with Strain.

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[14] A. W. Skempton, "The $\phi = 0$ Analysis of Stability and Its Theoretical Basis," *Proceedings*, Second International Conference on Soil Mechanics and Foundation Engineering, Rotterdam, Vol. 1, p. 72–78 (1948).

[15] Definitions of Terms and Symbols Relating to Soil Mechanics, (D 653 – 58), 1958 Book of ASTM Standards, Part 4.

solidation and creating pore pressures in the undrained specimen. If the variation in principal stresses, both total and effective, for specimen 96–4–4 are examined in Fig. 11, it is seen that while the principal stress difference in each case is the same at any particular degree of strain, the effective stresses are steadily decreasing. If effective stresses govern shearing resistance, how then can this resistance be maintained approximately constant while the effective stresses decrease? To explain this, the author has used the same concept as stated by Mr. Burmister, that "cohesion is really only a memory of a previous prestressed friction state."

Mr. Schmertmann shows arithmetically what the author attempted to demonstrate graphically in Fig. 8, namely, that the development of porewater pressure has little influence on effective stresses during application of the first half of the maximum deviator stress. The author attaches no significance to the pore-pressure behavior during the test in attempting to separate friction from cohesion. All reasoning is based on computed effective stresses, assuming that the total stresses and pore pressures can be measured with reasonable accuracy.

It is fortunate though that effective stresses remain relatively constant during initial loading, as this leads to the deduction that a frictional resistance is steadily developed due to strain on the potential failure plane. The author does not agree with Mr. Schmertmann that ϕ_e must be zero at zero strain. ϕ_e is a property of the material that requires shear strain to develop it. The factor of safety was introduced to try to take account of the degree to which friction is developed with stress application. The author has published curves showing the development of computed ϕ' from 0 to 35 deg as a function of strain (**10**). Of course ϕ' should not be a variable, but the curves were used to illustrate the need to agree on a reasonable failure criterion that is compatible with strains *in situ*. To be precise, the curves of Figs. 6 and 7 should have been broken at very low strains as are the curves of Fig. 5.

The author suggests that all shearing resistance is of a frictional nature and reserves the cohesion term for that portion of the frictional resistance which results from the "memory" of previous stresses and appears to be independent of applied effective stresses. These stresses are considered to be "locked-in" but are thought to decrease with time. Cohesion is attributed therefore to prestress with a superimposed time factor. Only the maximum possible cohesion is related to void ratio which is in turn dependent on stress history. To this extent the author's concept of cohesion differs from that of Hvorslev.

The unusual behavior of this soil is that it can maintain a particular shearing resistance while the effective stresses on the failure plane are reduced by more than one-half. The author suggests that an effort was made to "learn the reason for this behavior and then, if possible, relate it to a carefully defined friction or cohesion component." The success of this reasoning is definitely a matter of opinion.

It is true that the development of pore pressure up to one half maximum deviator stress has relatively little effect on effective stresses; $\bar{\sigma}_3$ is reduced by only a little more than 10 per cent. Up to this point, then the test is almost a drained test, and since the effective stress on the critical plane is so high, the computed ϕ' angle is little affected by the small pore pressure change. It is the pore pressure change during application of the second half of the deviator stress that is important. During this period, $\bar{\sigma}_3$ is reduced by about 60 per cent. The question is: Does this drastic change in effective stress under applied deviator stress occur in the field in the same manner? The author's contention is that it does not, that it is a test phenomenon, and that the interpretation and extrapolation of the laboratory test result should be given special consideration.

By introducing the concept of factor of safety, the author has, in effect, inferred a straight-line stress-strain relationship which is shown in the accompanying Fig. 11 together with the test curve for specimen 96-4-4. Which curve more closely represents the relation between stress and strain *in situ*? Which curve should be used to compute c' and ϕ'?

The author suggests that the actual relationship is closer to the "idealized curve" than to the "test curve." This is not to say that the "idealized curve" represents exactly what happens in nature but to suggest that it is the best approximation until very near to failure. This is a judgment factor based on the knowledge that the stress-strain curve is displaced more and more from the "idealized curve" as sample disturbance increases and on the belief that slope creep is not a dominant property of these brittle, sensitive clays. Failure is usually sudden and catastrophic, whereas creep occurs slowly and under drained conditions. This would result in consolidation and increased strength.

Referring to specimen 96-4-4, it is noted that at a strain of 0.2 per cent a deviator stress of 1.75 kg per sq cm has been applied and pore water pressure is 0.55 kg per sq cm. Over this stress range, both stress and pore water pressure are almost exactly proportional to strain. Pore pressure parameter A is equal to one third, exactly what it should be according to elastic theory. On further loading the proportionality of stress and strain does not apply but the increase in pore pressure continues to be proportional to strain until about 0.4 per cent strain. At this point, about 70 per cent of maximum deviator stress has been applied, and there is a drastic change in the ability of the specimen to resist the increase of major principal effective stress. It could be argued that the specimen had failed at less than 0.5 per cent strain. High A values are obtained in the laboratory only after large strains, and it is questionable if they have any practical significance in this material.

The critical question concerning shearing resistance is whether fundamental cohesion and friction parameters exist. Can strength be divided into two components, one component depending on applied effective stress and a second component independent of applied effective stress? Mr. Schmid thinks this cannot be done and is in favor of discarding the Mohr-Coulomb failure theory. This paper emphasizes the difficulty of interpreting tests on sensitive clay in terms of the Mohr-Coulomb theory, but in the author's view, this theory has too many advantages to warrant its discard at the moment. There is reason to believe that careful study of deformation in terms of effective stresses will result in satisfactory understanding of shearing resistance in clay in terms of the Mohr-Coulomb theory.

QUANTITATIVE DETERMINATION OF SOIL MONTMORILLONITE BY X-RAY DIFFRACTION*

By George R. Glenn[1] and R. L. Handy[2]

Synopsis

Montmorillonite exerts a strong influence on soil properties. X-ray diffraction offers the best qualitative method for clay mineral identification because *d*-spacings are more or less specific for a particular mineral. Other tests are affected to some extent by all clay minerals present, and individual contributions from each mineral are difficult to assign.

Unfortunately, X-ray diffraction intensities depend not only on the percentage of the reflecting crystals present but also on the absorption of X-rays by the sample and the amount of preferred orientation of the crystallites. Therefore special procedures are used to obtain random orientation in the sample holders. To correct for absorption caused by variations in sample packing and changes in composition, an internal standard was added and soil sample was mixed mechanically, and ground for 30 min. Diffraction intensities were then compared to those from the standard. The standard chosen was tetramethyl ammonium stannic chloride which has a strong diffraction at 7.43 Å, near to but not conflicting with those of the measured minerals.

Calibration curves for montmorillonite and quartz are presented and percentages of these constituents are determined for the *A*, *B*, and *C* horizons of Iowa Wiesenboden, Brunizem, Gray-Brown Podzolic, and Planosol soils. The montmorillonite contents are discussed in relation to plasticity, gradation, glycol retention, and cation exchange data.

Montmorillonite contents of air-dry, natural soils can be measured using X-ray diffraction with an internal standard, employing a correction for intensity variations due to interlayer water configuration. Correlations of per cent montmorillonite with plasticity index and with glycol retention are about on a par with analogous correlations between these data and 2-μ clay contents. Correlation of cation exchange capacity with per cent montmorillonite is better than that obtained with 2-μ clay.

X-ray diffraction offers the best qualitative method for mineral identification in soils because *d*-spacings are more or less specific for a particular mineral. Unfortunately, X-ray diffraction intensities depend not only on the percentage of the reflecting crystals present but also on absorption of X-rays by the sample and the degree of preferred orientation of reflecting crystallites. Absorption error may be corrected by use of an internal standard; a special sample mounting technique may be used to minimize preferred orientation.

A standard Geiger counter diffractometer geometry was adopted. Chart peak areas above background rather than peak heights were measured because of possible influences of variable peak broadening due to factors of crystallite size and lattice imperfections.

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* Presented at the Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

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Calibration curves using an internal standard may be prepared by analyzing samples of known mineral content. In this study quartz and montmorillonite were regarded as the two most important minerals present, and suitable curves were prepared for the (101) and (001) peaks, respectively. A soil montmorillonite was used for the standard curve. Glycolation of the montmorillonite was found to increase problems of preferred orientation, so air-dry samples were used. An investigation was made relating montmorillonite intensity to moisture content and mean (001) spacing and to study the need for a correction factor.

As a check on the reliability of the X-ray method, several soil samples were analyzed. Since there is no other direct method for measurement of the percentage of montmorillonite in soil, the X-ray data were correlated with various properties such as plasticity, gradation, glycol retention, and cation exchange capacity, all known to be strongly influenced by the amount of montmorillonite clay mineral. For comparison, the same properties were correlated with 2-μ contents of the same soils.

No attempt was made to correct for intensity variations due to isomorphous substitution in the montmorillonite lattice, which can be evaluated only by elaborate purification and chemical analysis. The X-ray study was conducted, recognizing that this factor could reduce the method to a semiquantitative analysis (**1**),[3] but with the feeling that isomorphous substitution would not be sufficient to invalidate the general findings of the research. To provide a rigorous test, the soil samples selected represent *A*, *B*, and C horizons of a Brunizem, a Gray-Brown Podzolic, a Wiesenboden, and a Planosol, all developed on Cary age glacial till in Iowa. The predominant mineral in all the samples was montmorillonite, with no appreciable 14-Å mineral appearing after glycolation.

USE OF INTERNAL STANDARD

The development of a completely standardized technique for the quantitative analysis of soil mixtures is a most important consideration. The indirect analysis approach using an internal standard is dictated by the fact that the X-ray absorption properties of different soils are variable, unknown, and different from that of montmorillonite. The use of the internal standard corrects for variations in absorption (**2**).

An internal standard must give few, strong diffraction lines close to but not conflicting with lines from the minerals to be measured. In addition, the crystal habit should avoid tendencies toward preferred orientations in the sample holder. These conditions are best met by the use of a standard that crystallized in the cubic system.

For clay mineral work the standard should have an unusually large *d*-spacing. The standard chosen was tetramethyl ammonium stannic chloride, which is face-centered cubic with a very strong (111) reflection at 7.43 Å (**3**). This chemical is easily prepared in the laboratory (Appendix I[4]) (**4**). Disadvantages are a tendency to react with calcium carbonate when present in a moist soil, and a relatively high mass absorption coefficient for copper Kα radiation, 122 as compared to 35 for quartz. Fortunately a relatively small amount of internal standard was needed to give peaks comparable in area to those from major minerals in soil. A constant amount of the standard, 0.091 g, was added to each gram of soil for analysis and to known mixtures of minerals plus diluent for preparation of calibration curves. Reaction with calcium carbonate was avoided with calcareous soils by analyzing immediately after mixing and with the soil air dry.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

[4] See p. 1286.

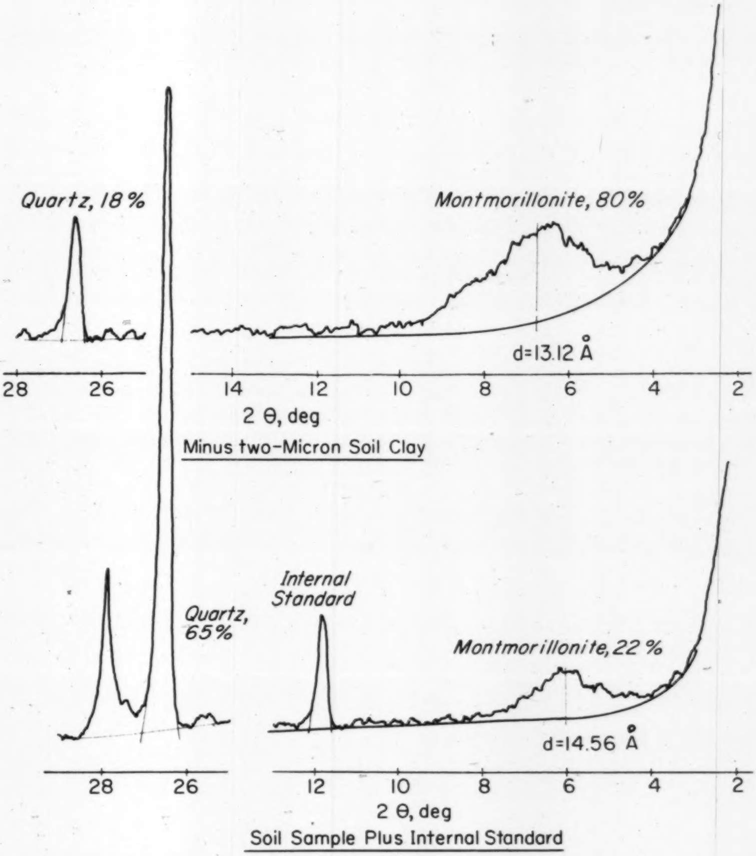

Fig. 1.—Typical X-ray Diffraction Patterns, Showing Location of Baseline for Measurement of Area and Calculation of Mean *d*-spacing.

X-ray Diffraction Procedures

Sample Preparation:

All X-ray samples were air dried and quartered to reduce the sample size and mixed and ground by a motor-driven mortar and pestle for 2 hr and hand ground in a small agate mortar until all passed a No. 270 sieve. The measured amount of internal standard was then blended with each sample by means of a 30-min grinding period. In most instances, three slides of each sample were prepared for X-ray analysis. Montmorillonite and other clay minerals, being sheet-like, show a tendency toward preferred orientation in a sample holder. Although preferred orientation is useful for increasing intensities from the basal spacings, it was found that it is not readily reproducible. Preferred orientation was avoided in the present study by use of open sample holders loaded from the back (5). Sample holders are Brown & Sharp 12-gage aluminum with a $\frac{3}{4}$-in. hole. For loading, the holder is taped face down on a glass slide, and material is sifted in from the back, compacted with the edge of a spatula, and struck off flush with a razor blade. After additional material is sifted on, the sample is compacted with a piston load of 75 g and again struck off. A glass slide is taped to the back for additional support, and the front slide is untaped and carefully lifted to expose the sample.

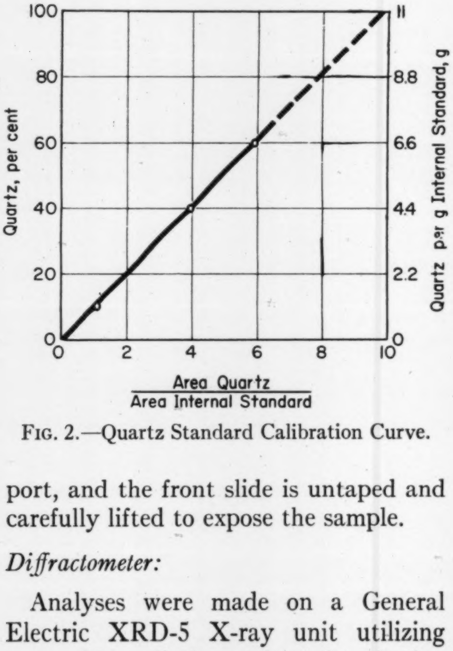

FIG. 2.—Quartz Standard Calibration Curve.

Diffractometer:

Analyses were made on a General Electric XRD-5 X-ray unit utilizing copper Kα radiation and a 4-deg viewing angle, 1-deg beam slit, MR soller slit, 0.2-deg detector slit, 2 deg (2θ) per min scan rate, 3-sec time constant, 1 in. per min chart speed, and a linear ratemeter response of 2000 cps for a full-scale deflection.

The crystallite size of montmorillonite is fine enough to cause considerable broadening of the diffraction line, particularly in the basal reflections because of the plate-like nature of the clay particles. Therefore diffraction peak area instead of peak height was used as a measure of intensity. The peak areas were measured by planimeter and represent the total count above a background line drawn as a smooth curve through the mean of the low points adjacent to the peak. Illustrative background lines are sketched in Fig. 1.

Because the montmorillonite basal *d*-spacing is variable, the mean *d*-spacing was determined by a vertical line through the diffraction peak centroid, splitting the peak area in half (Fig. 1).

CALIBRATION CURVES

Quartz:

A standard curve for measurement of the amount of quartz in soil was determined from known mixtures of quartz plus diluent plus internal standard. The quartz reference was crushed Ottawa sand separated by water elutriation to obtain 5 to 10-μ size material. Alpha alumina (Al_2O_3) was used as diluent. The calibration curve for the strongest quartz line, d_{101} = 3.35 Å, is presented in Fig. 2.

Montmorillonite:

A reference sample of soil montmorillonite[5] was obtained by dispersing in distilled water a noncalcareous *C*-horizon loess sample (sample 46-1) from the Marshall Series in Page County, Iowa, and pipetting aliquots to obtain representative minus 2-μ material. The cation exchange capacity of this clay is 57.4 meq per 100 g (6) with calcium the major adsorbed cation and minor sodium and potassium.

X-ray analysis showed the soil clay to be montmorillonite and quartz, with no other recognizable peaks. Use of internal standard and the quartz calibration curve (Fig. 2) indicated that the quartz content was in the neighborhood of 18 per cent. For preparation of the montmorillonite calibration curve the montmorillonite content was therefore taken as about 80 per cent. The initial calibration curve for air-dry untreated montmorillonite (001) peak areas was prepared in the same manner as that for quartz, by adding measured amounts of Al_2O_3 diluent and the internal standard.

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[5] b_0 = 8.98 Å, from (060) peak.

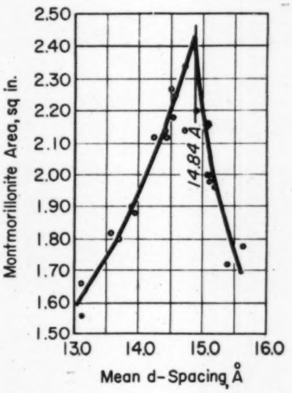

FIG. 3.—Montmorillonite Area Versus *d*-spacing.

Samples: 80 per cent soil-montmorillonite with variable water content.

Data points: Areas measured above background of diffraction pattern, recorded at 2000 cps and 1 in. per min. Mean *d*-spacings determined from centroids of areas.

The montmorillonite calibration curve showed considerable point scatter, perhaps related to the variable moisture content. Several paths were open: to glycolate the samples or control their moisture contents to try to obtain some structural uniformity in the interlayer; or to relate intensity to moisture content or *d*-spacing so as to develop a correction factor.

Glycolation, although ostensibly the simplest remedy, introduced other problems. Much difficulty was experienced in obtaining random orientation; once random orientation was obtained the montmorillonite peak heights were reduced to about one half and the areas to about one fourth at the values obtained with air-dry samples. Glycolated samples containing calcite reacted unfavorably with the internal standard, which is at home in an acid environment.

To check on the possibility of a systematic relationship of peak intensity to moisture content, water was added manually to the air-dried mineral, and 24 X-ray patterns were made without the addition of an internal standard. The montmorillonite peak areas were found to vary depending on moisture content, reaching a maximum when the interlayer spacing was approximately 14.8 Å (Fig. 3) and the water content was between 8 and 9 per cent.

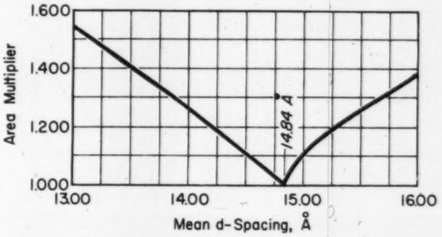

FIG. 4.—Montmorillonite Area Multiplier Versus *d*-spacing.

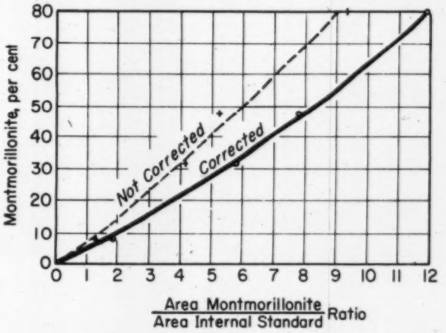

FIG. 5.—Standard Calibration Curve for Soil Montmorillonite.

Based upon the relationship of Fig. 3, an area correction curve was drawn as shown in Fig. 4. Area data for the montmorillonite calibration curve were corrected by noting the mean *d*-spacings and multiplying by the appropriate correction factors from Fig. 4. The montmorillonite calibration curves before and after correction are shown in Fig. 5. That this adjustment is valid is shown by the reduced point scatter for the corrected curve.

An implication of the diffraction area maximum occurring at a *d*-spacing of 14.8 Å is that this may represent a maximum state of ordering of interlayer water. This also is reflected in increased peak sharpness as this interlayer spacing is approached. The (001) distance from a sample heated to 300 C for 2 hr was found to be 10.0 Å, indicating an effective interlayer spacing of 4.8 Å for water. Several configurations for interlayer water in calcium montmorillonite are suggested by Barshad (7); the 4.8 Å is in the proper range for a double layer but does not precisely coincide with any of the structures hypothesized.

SOILS

The first application of the X-ray montmorillonite analysis method was to study horizons of modal Brunizem, Wiesenboden, Gray-Brown Podzolic, and Planosol profiles developed on late Wisconsin (Cary) glacial till in central Iowa. Soil samples were made available by the Department of Agronomy, Iowa State University, and use was made of available particle size and cation exchange data (8).

TABLE I.—SUMMARY OF FINDINGS.

Great Soil Gp., Series, & Profile No.	Horiz., Texture Depth	Point No.	Soil Fraction %			C.E.C. Me./100g	Glycol Ret. Mg /gm	% By X-ray Diffraction		Atterberg Lim Air Dry Soil		
			Sand[a]	Silt[b]	Clay[c]			Qtz	Montm.	L.L.	P.L.	P.I.
Brunizem	A lo 0-8in.	1	39	36	25	26.7	62.9	43	9	40	30	10
Clarion	B lo 16-20in.	2	43	31	26	21.6	67.2	40	11	37	20	17
P-97	C_2 lo 32-36in.	3	38	44	18	41.9	61.5	40	11	35	20	15
Wiesenboden	A cl lo 0-8in.	4	26	39	35	43.1	77.0	35	13	54	39	15
Webster	B cl lo 17-21in.	5	33	33	34	30.6	89.9	65	22	42	26	16
P-137	C_2 lo 31-37in.	6	40	36	24	33.3	77.2	28	18	39	24	15
Podzolic	A_2 si lo 3-7in.	7	30	52	18	12.8	41.0	38	8	25	20	5
Hayden	B_{21} cl lo 16-21in.	8	32	34	34	23.6	91.8	39	19	47	23	24
P-401	C_2 lo 36-42in.	9	34	44	22	32.1	67.5	38	18	33	24	9
Planosol	A_{21} si lo 4-9in.	10	27	55	18	13.1	37.4	48	3	25	22	3
Ames	B_{22} cl 20-24in.	11	34	26	40	25.9	92.5	48	22	47	25	22
P-568	C_2 sa cl lo 40^+ in.	12	47	28	25	34.4	45.6	45	16	27	16	11

a. Sand - Particle sizes > 50μ. b. Silt - Particle sizes 2 through 50μ. c. Clay - Particle sizes < 2μ.

Notes: 1. Texture-U.S.D.A. System, lo-loam, cl-clay, si-silt, sa-sandy. 2. Soil fraction, C.E.C. data after Cardosa (8)

In most instances three samples of each soil were mounted and X-rayed. Quartz (101), montmorillonite (001), and internal standard (111) diffraction peak areas were measured. The ratio of each quartz area to area of the internal standard was computed, and the percentage of quartz was read from Fig. 2, using the average ratio from two or three determinations. The mean *d*-spacing for each montmorillonite area was established, from which a multiplier was determined using Fig. 4; this multiplier was applied and the adjusted area was expressed as a ratio to that of the internal standard, and the percentage of montmorillonite read from Fig. 5, using the average of two or three determinations. The measured percentages of montmorillonite and quartz are shown in Table I.

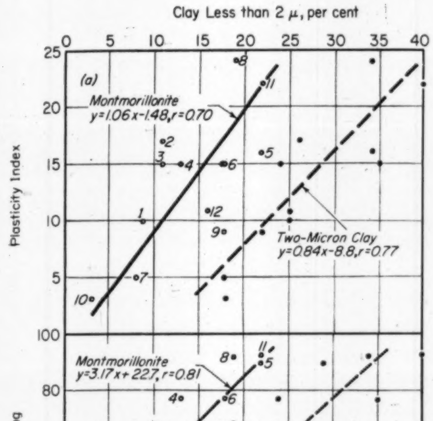

FIG. 6.—(*a*) Plasticity Index Versus per cent Montmorillonite, (*b*) Per Cent Montmorillonite Versus Glycol Retention, and (*c*) Per Cent Montmorillonite Versus Cation Exchange Capacity.

Open circles indicate montmorillonite; closed circles indicate less than 2-μ clay.

CORRELATIONS

Montmorillonite is believed to be the primary mineral influencing many soil properties; therefore, plasticity index data, ethylene glycol retention data, and cation exchange capacities were plotted against montmorillonite contents in Figs. 6(*a*), (*b*) and (*c*) respectively, and the coefficients of correlation calculated. For comparison the relationships between these properties and the percentage of minus 2-μ clay are presented in the same graphs with their coefficients of correlation. Because both x and y values are measured subject to error, the best fits are obtained when the curves and equations describe the bisector of the acute angle between lines of regression of x on y and y on x, determined by the method of least squares.

Plasticity Index:

Figure 6(*a*) shows a graph relating plasticity index[6] to the percentage of montmorillonite and of minus 2-μ clay[7] (Table I). For plasticity versus montmorillonite, the equation believed to best describe the data is

$$y = x - 1.5,$$

the coefficient of correlation r being equal to 0.70. A value of ± 1.0 indicates perfect correlation and a value of 0 indicates no correlation.

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[6] The plasticity index was determined by ASTM Method of Test for Liquid Limit of Soils (D 423 - 59 T) and ASTM Method of Test for Plastic Limit and Plasticity Index of Soils (D 424 - 59), 1958 Book of ASTM Standards, Part 4.

[7] Clay content data for the Iowa soils are after Cardosa (**8**), who used a pipet method. Duplicate 10-mg samples were treated with hydrogen peroxide to destroy organic matter and placed on an end-over-end shaker for 24 hr, using sodium hexametaphosphate as dispersing agent. The less than 2-μ clay was determined with a 25-ml pipet at 10 cm depth, using the proper time interval according to Stokes' law.

Similarly, the best fit for plasticity index versus 2-μ clay content is

$$y = 0.84\,x - 8.8,$$

with r equal to 0.77.

Slopes of the two lines indicate that, in general, plasticity index is influenced more by the montmorillonite content than by the total 2-μ clay because of dilution with other less plastic minerals (9). The x intercepts suggest that a minimum montmorillonite content of 1.5 per cent or a minimum total clay content of about 10 per cent is required for these soils to indicate a plasticity index; below these limits the soil will tend to be nonplastic.

TABLE II.—COEFFICIENTS OF CORRELATION.

Property	Correlation Coefficient, r, Versus	
	Montmorillonite Content	Clay Content
Plastic limit	0.07	0.49
Liquid limit	0.35	0.84
Plasticity index	0.70	0.77

The coefficients of correlation are comparable. Perhaps pertinent is the fact that the limits from which the plasticity index is calculated correlate better with clay content than with montmorillonite content, as shown in Table II.

Glycol Retention:

Upon exposure to a vapor of ethylene glycol, the interlayers of montmorillonite tend to absorb the molecules and expand to a uniform d-spacing. An equilibrium amount of ethylene glycol is retained under conditions of vacuum and with an available free glycol surface. Glycol retention was measured for the various soil samples by the method of Martin (10) as modified from Dyal and Hendricks (11). Data are given in Table I.

The curves of glycol retention versus montmorillonite content and clay content are shown in Fig. 6(b). The former may be approximated by the equation

$$y = 3.2\,x + 23.$$

The correlation coefficient is 0.81. By extrapolation of the curve, the y intercept indicates that the glycol retention for a similar soil with no montmorillonite would be about 23 mg per g. A value of 31 mg per g has been reported for pure muscovite (10). Similarly, extrapolation to 100 per cent montmorillonite gives a glycol retention value of 340 mg per g, compared with values of 260 to 295 for various bentonites (10).

The equation for glycol retention versus per cent clay (Fig. 6(b)) is

$$y = 2.8\,x - 5.6,$$

with a correlation coefficient of 0.84. Thus, as in the case of plasticity index, glycol retention is influenced more by montmorillonite content than by total clay, but is related to either about equally well.

Cation Exchange Capacity:

Cation exchange capacities in Table I represent the total of exchangeable hydrogen determined by the barium acetate method plus calcium, magnesium, and potassium removed by treatment with a neutral ammonium acetate solution.[8] Data are given in Table I. Calcium dominated exchange positions in all soil samples.

Plots of cation exchange capacity versus percentages of montmorillonite and of 2-μ clay (Fig. 6(c)) show considerable point scatter. The correlation coefficient for cation exchange capacity versus clay content is only 0.24, and no curve is shown. A better correlation was found between cation exchange capacity and montmorillonite content. The estimated equation for the curve is

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[8] Cation exchange capacity data are from Cardosa (8).

$$y = 1.4\,x + 8.3.$$

Extrapolation of this curve gives a cation exchange capacity of 8.3 meq per 100 g for similar soil with no montmorillonite, and a cation exchange capacity of 150 for 100 per cent montmorillonite; however, little significance should be attached to these values because of the low coefficient of correlation, $r = 0.40$.

2 μ Clay Content:

The correlation coefficient for per cent montmorillonite versus 2-μ clay is 0.70. The equation obtained as before, assuming random errors in both measurements, is $y = 0.8x - 7.2$. Considering clay content as the independent variable, the line of regression of y on x is $y = 0.56x - 0.74$, or almost through the origin. Forcing the curve through the origin gives $y = 0.53x$. Data are too limited and points too scattered to determine which is the most appropriate equation. The latter would indicate that average clay fractions of central Iowa soils are about one-half montmorillonite, whereas the former would indicate that the proportion of montmorillonite in the clay fraction tends to be progressively higher in more clayey soils, that is, B horizons.

CONCLUSIONS

1. Montmorillonite content of air-dry natural soils can be measured by X-ray diffraction with an internal standard, employing a correction for variation of intensity due to configuration of interlayer water.

2. The correlations of percentage of montmorillonite with plasticity index and with glycol retention are about on a par with analogous correlations between these data and 2-μ clay contents by pipet analysis. The correlation of cation exchange capacity to percentage of montmorillonite is considerably better than the correlation with 2-μ clay content.

Acknowledgments:

This work was carried on under: (1) "X-ray Diffraction Analysis of Highway Materials," Project HR-48 of the Iowa Highway Research Board, supported by funds from the Iowa State Highway Commission; (2) 1960 Summer Institute for Research Participation, supported by Iowa State University under grants from the National Science Foundation; and (3) Special Research Project at Southern Illinois University entitled "Quantitative Determination of Soil Montmorillonite by X-ray Diffraction," supported by funds from the University.

Grateful acknowledgment is made to F. F. Riecken, Department of Agronomy, Iowa State University, for making soil profile samples available.

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APPENDIX I

PREPARATION OF TETRAMETHYL AMMONIUM STANNIC CHLORIDE

A 10 per cent solution of tetramethyl ammonium hydroxide is titrated to neutrality with dilute (about five per cent) hydrochloric acid, giving the following reaction:

$$(CH_3)_4NOH + HCl \rightarrow [N(CH_3)_4]Cl + H_2O$$

Addition of anhydrous stannic chloride liquid forms tetramethyl ammonium stannic chloride crystals, according to the following equation:

$$2[N(CH_3)_4]Cl + SnCl_4 \rightarrow [N(CH_3)_4]_2 \cdot SnCl_6$$

APPENDIX II

X-RAY DIFFRACTION PATTERN DATA FOR SOILS

Soil Profile No. & Depth, in.	Graph Plot No.	Montm. Area, A_M	Mean d-Spacing	Adjusted A_M	Internal Std Area, A_M	A_M/A_{IS} Ratio	$\bar{X}$ Of Ratio	Quartz Area A_Q	A_Q/A_{IS} Ratio	$\bar{X}$ Of Ratio
P-97 (0-8)	1	0.54 0.52 0.44	14.13 14.24 13.95	0.654 0.614 0.555	0.33 0.35 0.33	1.98 1.75 1.68	1.80	1.41 1.33 1.54	4.27 3.80 4.66	424
P-97 (16-20)	2	0.70 0.70 0.64	14.53 14.13 14.59	0.770 0.847 0.700	0.36 0.33 0.40	2.14 2.54 1.75	2.15	1.43 1.26 1.64	3.97 3.82 4.10	3.96
P-97 (32-36)	3	0.56 0.62 0.55	14.40 14.72 14.67	0.640 0.650 0.580	0.28 0.29 0.30	2.29 2.24 1.93	2.15	1.08 1.06 1.29	3.86 3.66 4.30	3.94
P-137 (0-8)	4	0.73 0.82 0.79	14.56 14.72 14.81	0.797 0.854 0.804	0.33 0.33 0.32	2.42 2.55 2.51	2.49	1.08 1.07 1.28	3.27 3.24 4.00	3.50
P-137 (17-21)	5	0.66 0.61 0.59	14.24 14.56 14.60	0.779 0.664 0.638	0.17 0.20 0.15	4.58 3.32 4.26	4.05	1.10 1.09 1.09	6.47 5.45 7.53	6.48
P-137 (31-37)	6	0.64 0.80 0.81	14.56 14.72 14.72	0.698 0.840 0.843	0.21 0.27 0.25	3.38 3.14 3.40	3.31	0.66 0.72 0.68	3.19 2.68 2.74	2.87
P-401 (3-7)	7	0.48 0.47 0.52	1437 1417 1448	0552 0568 0577	038 032 0.36	1.45 1.77 1.60	1.61	1.52 1.23 1.29	4.00 3.84 3.58	381
P-401 (16-21)	8	0.88 0.90 0.89	14.32 14.60 14.38	1.020 0.972 1.018	0.31 0.27 0.28	3.29 3.60 3.63	3.51	1.20 1.06 1.09	3.87 3.93 3.89	3.90
P-401 (36-42)	9	1.04 0.85 0.84	14.42 14.60 14.48	1.175 0.919 0.932	0.34 0.28 0.28	3.45 3.28 3.33	3.35	1.26 1.02 1.14	3.71 3.64 4.07	3.81
P-568 (4-9)	10	0.18 0.20	14.13 14.48	0.218 0.222	0.30 0.33	0.727 0.674	0.70	1.58 1.45	5.27 4.38	482
P-568 (20-24)	11	0.81 0.78 0.81	14.32 14.36 14.52	0.940 0.900 0.900	0.23 0.23 0.23	4.09 3.91 3.91	3.97	1.12 1.13	4.88 4.91	489
P-568 (40+)	12	0.50 0.52	14.40 14.48	0.568 0.568	0.20 0.19	2.84 3.18	3.01	0.97	5.24	524

DISCUSSION

Mr. J. E. Mitchell (*by letter*).[1]—The authors are to be commended for their contribution to the complex problem of clay mineralogy and especially the quantitative determination of montmorillonite. It would have been interesting had the authors made a correlation between montmorillonite content as determined by Differential Thermal Analysis (DTA) and X-ray diffraction. DTA has somewhat the same difficulties as X-ray diffraction in effect of particle size, adsorbed cations, adsorbed water, etc., but when applied to the limited number of samples the authors used, these effects would have been more or less constant throughout the series, and a good correlation might have been obtained. A better correlation might have been obtained from the plasticity index (PI) had the tests been made on soil which had not been allowed to air-dry. Studies at the Waterways Experiment Station indicate that the values for the liquid limit of a soil containing montmorillonite change with the elapsed time after the addition of water to the air-dry soil. Unless this elapsed time of rehydration is kept constant for each test, the liquid limit values are often erratic.

Messrs. George R. Glenn and R. L. Handy (*authors' closure by letter*).—We sincerely appreciate Mr. Mitchell's evaluation and would like to add that indeed in the late phases of the investigation parallel studies were undertaken with DTA, first with bentonites and later with the soil samples used for X-ray diffraction. Excellent results with the calcium and sodium bentonites[2] indicate a satisfactory DTA methodology, but results with the soil samples were not so impressive.

Prior to DTA, all samples were kept four days *in vacuo* over a solution which gave 50 per cent relative humidity at room temperature. First, endothermic peak areas, characteristic of the sorbed water, were measured. Due to depletion of samples only eight of the soils were tested, so correlation coefficients are rather poorly estimated.

The r for DTA peak area versus plasticity index (PI) is -0.59, indicating a negative slope to the curve and a very poor relationship. However, r for DTA versus plastic limit is 0.81, higher than for either X-ray or clay content analysis. The r for DTA versus glycol retention is 0.39, which is lower than $r = 0.90$ from X-ray analysis of the same eight samples. Therefore DTA of prehumidified natural soils does not appear to give as good a quantitative measure of soil montmorillonite as X-ray diffraction, probably due to variability of the adsorbed cations. Pretreatment to obtain monoionic soils and remove organic matter would probably improve correlations.

Changes in liquid limit with elapsed time after wetting are also the rule with Iowa montmorillonitic soils; therefore soils were wet above the liquid limit and dried down for the tests.

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[1] Chief, Soils Test Section, Embankment and Foundation Branch, Soils Div., U. S. Army Engineer Waterways Experiment Station, Vicksburg, Miss.

[2] J. G. Laguros, L. L. Reign, and R. L. Handy, "Effect of Exchangeable Calcium on the Low Temperature and Endothermic Reaction of Montmorillonite," to be presented at the Forty-First Annual Meeting of the Highway Research Board, Jan. 8–12, 1962.

A STUDY OF ARTIFICIAL SOILS*

By E. T. Selig[1] and R. D. Rowe[1]

Synopsis

A study of artificial soils was undertaken, the purposes of which were to determine (1) the variety of properties that can be obtained with artificially prepared soils, (2) the reproducibility and stability of these properties, and (3) the extent to which these properties resemble those of natural soils. Soils synthesized from clay minerals, non-clay soil minerals, and water, ethylene glycol, or mineral oil in many different combinations were examined using standard soil identification procedures: (1) Atterberg limits, (2) Harvard miniature compaction, (3) unconfined compression, and (4) triaxial compression. It is concluded that artificial soils may be prepared which exhibit a wide variety of natural soil properties that are reproducible and stable. For general laboratory studies not requiring a specific natural soil, particularly where a standard soil is desired for purposes of comparison, the use of artificial soils appears promising.

The solutions of many basic engineering problems involving soils require a knowledge of soil behavior that may often best be gained through model studies and other laboratory investigations. Among such problems of current interest are the determination of pressures on buried structures, the prediction of the response of foundations to dynamic loads, the analysis of off-the-road vehicle performance, and the understanding of stress wave propagation in soils. Rather than pertaining to specific soils, these problems usually are general in scope; hence, it is desirable to use several representative soils in the investigations. Further, the properties of the test soils must be controllable, reproducible and consistent. Obtaining a variety of natural soils is difficult, but even if the natural soils were readily available, they would not, in general, meet the above criteria. In view of these difficulties, the study discussed in this paper was undertaken to determine if the characteristics of natural soils could be satisfactorily simulated in the laboratory by artificial soils which would be homogeneous, adequately controllable, and possess reproducibility and stability over a range of properties.

In engineering problems involving soils, many widely different soil properties—permeability, plasticity, consolidation, swelling, shear strength, density, stiffness—are encountered. The importance of each of these in a given problem depends upon the nature of that problem. It is improbable that one could synthesize a "universal" soil capable of exhibiting any range of property values prescribed to suit a given problem. It is probable, however, that one could synthesize a group of soils capable of exhibiting the range of values of those soil properties most important to a given problem, with other properties unspecified. The purposes of this present study have been to determine (1) the variety of properties that can be obtained with artificially prepared soils, (2) the reproducibility ond stability of these properties, and (3) the extent to which these properties resemble those of natural soils. First, a search was conducted for ingredients which could be used to create artificial soils having the desired properties. Attention primarily was focused on materials present in natural soils, although other materials were considered. Soils synthesized from many different combinations of the selected ingredients were then examined to determine their properties using several standard soil identification procedures: (1) Atterberg limits, (2) Harvard miniature compaction, (3) unconfined compression, and (4) triaxial compression. The findings of the search and typical test results with the artificial soils are discussed in the following sections of this paper.

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* Presented at the Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] Associate Research Engineer and Laboratory Engineer, respectively, Soil Mechanics Section, Armour Research Foundation, Chicago, Ill.

Among the many possible applications of artificial soils, there are a number which appear promising. Used as standard soils, they are a particularly valuable tool in design and development for comparing the effectiveness of various types of equipment, such as that for cutting, compacting and traversing soil. Comparative analyses of this type are frequently best carried out, especially in the preliminary stages, on small models under the carefully controlled laboratory conditions possible with artificial soils. The concept of a soil tank for evaluating the performance of off-the-road vehicles, analogous to wind tunnels for airplanes and towing tanks for ships, has been proposed by the Army Ordnance Land Locomotion Research Laboratory, where several types of artificial soils have already been used for this purpose (**4**).[2] The availability of several standard soil types should also provide the most satisfactory means of calibrating and comparing different types of soil testing equipment, for training laboratory personnel in the proper techniques of soil testing, and for teaching the fundamentals of soil behavior.

Although not called "artificial soils," a few types of these materials have already been used by many investigators in fundamental studies of soil behavior (**5,6**) and in model studies of various structural-soil interaction problems (**7,8**). One of the apparent limitations of the artificial soils is that the structure of undisturbed natural soils, built up by nature over a long period of time, cannot easily be duplicated, if at all. But many of their characteristics can equally well be studied with remolded soils and, in addition, many problems involve remolded soils, in which cases this limitation no longer exists. It seems reasonable to believe that if one prepares an artificial soil from entirely natural soil ingredients, it can be just as much a real soil as a natural remolded soil, the principal difference being the degree to which the material has been broken down before being remolded.

ARTIFICIAL SOIL INGREDIENTS

The principal ingredients of soils, taken as a whole, may be broadly classified into four groups: (1) clay minerals, principally montmorillonite, kaolinite, and illite, (2) non-clay minerals, such as silt, sand, and gravel, (3) organic matter, such as vegetation and its by-products, and (4) a liquid, which is primarily water, but which may contain a number of substances in solution. In this study, ingredients were selected to represent all of these groups except the third—organic matter. That they be prevalent in natural soils was not necessarily used as a criterion for selecting them because some of the individual characteristics of soils can be simulated by non-soil constituents. Except for liquid ingredients, however, only minor consideration was given to non-soil constituents in this study of artificial soils. Aside from the difficulty of finding suitable materials, there were two reasons for this: (1) a selection of suitable clay minerals, silts, and sands was found to be readily available, and (2) the uncertainty as to the validity of soil behavior predictions is greater for artificial soils made of non-soil constituents than for artificial soils made of natural-soil constituents.

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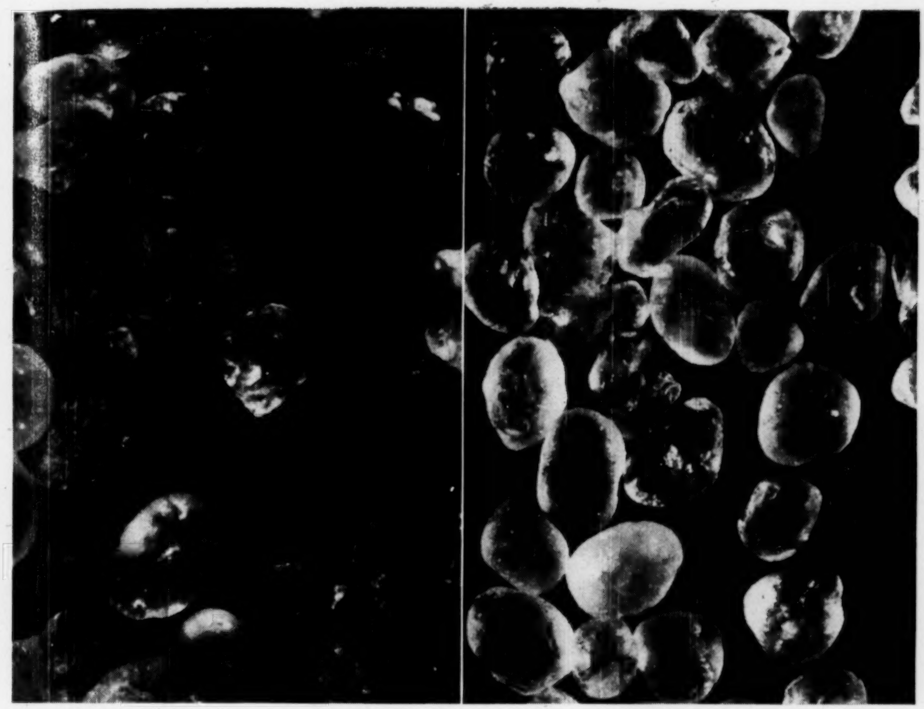
[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

TABLE I.—CHARACTERISTICS OF CLAY MINERALS.

Mineral	Volclay—also sodium, western, Wyoming, or S. Dakota bentonite.	Ohio H-W weathered bond B fire clay.	Florida plastic kaolin.	OM-4 Kentucky ball clay.
Supplier	American Colloid Co., Skokie, Illinois.	Harbison-Walker Refractories Co., Pittsburg, Pa.	Edgar Plastic Kaolin Co., Edgar, Florida.	Kentucky-Tennessee Clay Co., Mayfield, Ky.
Source	Wyoming or South Dakota.	South Webster, Ohio.	Edgar, Florida.	Kentucky old mine 4.
Mineral content	92–95 per cent sodium montmorillonite.	Primarily illite.	Better than 99 per cent kaolinite.	Approx. 75 per cent kaolinite — remainder illite, montmorillonite, feldspar, and quartz.
Preparation	Airfloated.	Washed, not airfloated.	Airfloated.	Airfloated.
Atterberg limits	LL = 437, PL = 47 PI = 390, SL = 6	LL = 39.0, PL = 21.0 PI = 18.0, SL = 13.4	LL = 70.2, PL = 40.8 PI = 29.4, SL = 22.8	LL = 62.6, PL = 31.3 PI = 31.3, SL = 21.6
Particle size	80–90 per cent smaller than 0.5 μ	94 per cent less than 105 μ	38 per cent less than 0.5 μ, 92 per cent less than 10 μ	
Specific gravity	2.90	2.75	2.66	2.69
Color	White when dry, light tan when wet.	Light gray when dry, darker when wet.	White.	Tan when dry, medium brown when wet.

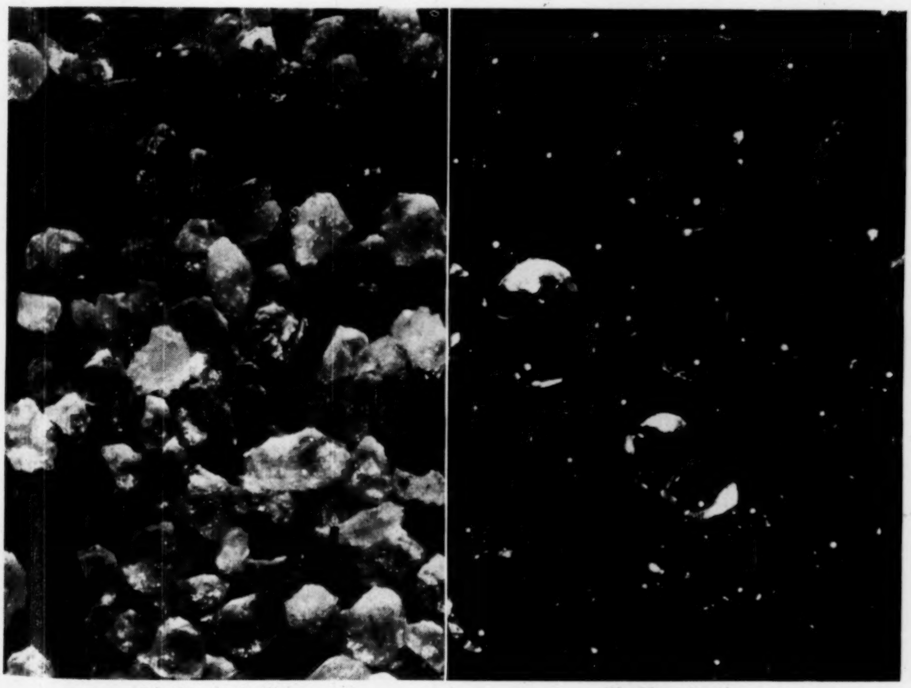
Four commercially available clay minerals were selected for this study to represent the three principal clay mineral groups: Wyoming bentonite is 92 to 95 per cent sodium montmorillonite, the fire clay is primarily illite, the kaolin is better than 99 per cent kaolinite, and the ball clay is a mixture containing 75 per cent kaolinite with the remainder illite, montmorillonite, and silicon dioxide. The descriptions of these materials are given in Table I.

Two types of sand were obtained for use in this study: a natural, whole-grained, silica sand from sandstone deposits in Ottawa, Ill., and a natural river sand from the Chippewa River, Wis. The particle size distributions for these materials are given in Fig. 3. The shape of the grains is shown by the photographs in Fig. 1. These may be compared to more angular crushed silica and to manufactured spherical glass beads shown in Fig. 2. A finely ground silica sand, normally referred to as silica flour, was ob-



(*a*) River sand. (*b*) Ottawa sand.

FIG. 1.—Natural Sands Magnified 20 Times.



(*a*) Crushed silica. (*l*) Glass beads.

FIG. 2.—Artificial Sands Magnified 20 Times.

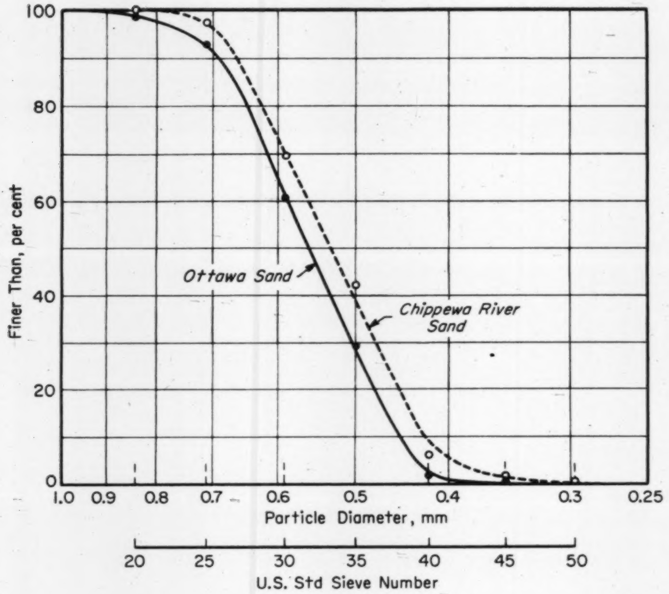


FIG. 3.—Grain Size Distribution of Ottawa and Chippewa River Sands.

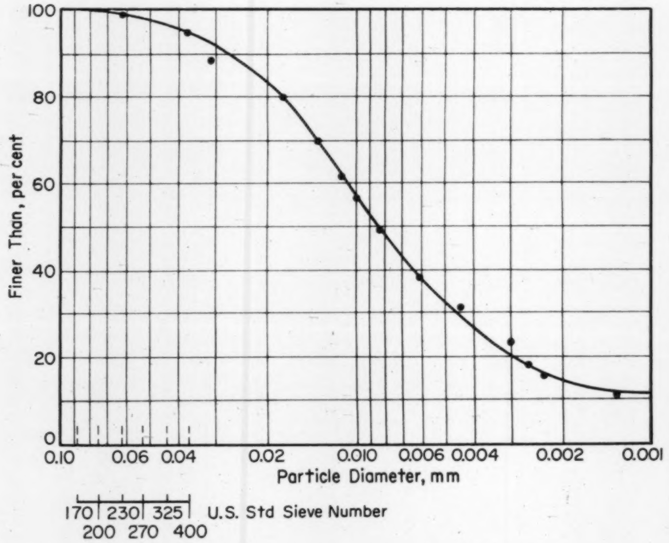


FIG. 4.—Grain Size Distribution of Silica Flour.

tained to represent silt. The particle size distribution of this material is given in Fig. 4.

In addition to water, two other liquid constituents were used in this study of artificial soils: ethylene glycol ($C_2H_6O_2$), specific gravity 1.113, and a DTE light mineral oil of paraffinic nature, specific gravity of 0.85.

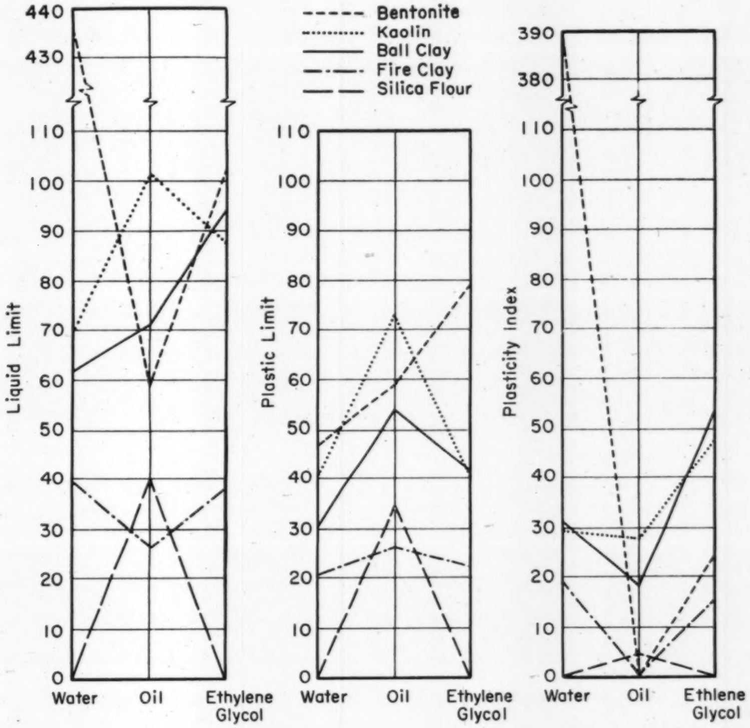


FIG. 5.—Comparison of Atterberg Limits for Soils Using Several Liquids (For Comparison, All Liquids Were Assigned a Specific Gravity of 1).

CHARACTERISTICS OF ARTIFICIAL SOILS

The artificial soils were prepared by mixing, in various proportions, the ingredients described. A few of the possible combinations have been selected to demonstrate the range of characteristics which can be obtained. Standard soil identification tests (1,2) were carefully used in evaluating these soils so that the characteristics could be most easily and meaningfully compared with those of natural soils.

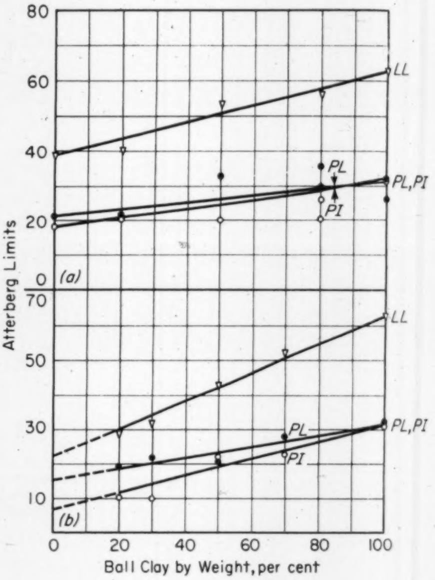
Atterberg Limits:

In this study, the liquid limit, plastic limit, and plasticity index were determined for each of the clay minerals alone and for the silica flour using water, ethylene glycol, and mineral oil as liquids. Atterberg tests were also performed on three-component mixtures using two solid ingredients and water. The purpose of these tests was to show how the indices vary with a change in per cent of each of the solid constituents.

The bentonite clay was the most difficult to work with. At its very high liquid limit (437), it had a smooth, soft, butter-like consistency. At low water contents, it was sticky and adhered strongly to the tools and dishes. Heat was generated by a chemical reaction when ethylene glycol was mixed with bentonite. Also, it was found that, when a mixture of bentonite and ethylene glycol was allowed to stand, some of the glycol separated. Therefore, this artificial soil was not a stable one. To a lesser extent the same thing occurred for mixtures of glycol with other clays.

The results of the tests for clays and silica flour alone mixed with each of the three liquids are shown graphically in Fig. 5. The silica flour was indicated to be non-plastic. Since the comparison of moisture contents of soils in which several liquids of different specific gravity are used cannot be meaningfully made on a weight basis, the quantities of the various liquids involved in the Atterberg limits were related to the corresponding quantities of water on a volume basis, and then expressed as percentages by weight for a hypothetical specific gravity of 1. The most striking change in plasticity occurred with bentonite when the water was replaced by the other liquids. With water, bentonite had a plasticity index of 390, but with ethylene glycol it was 23.4 and with oil it was essentially 0. In addition, oil added to fire clay gave a non-plastic mixture with a gritty consistency, almost like that of wet sand. When the silica flour was mixed with oil, both liquid and plastic limits were obtained and the mixture exhibited the small amount of plasticity characteristic of clayey-silts. With both water and ethylene glycol, however, the silica flour alone had the characteristics of a pure silt.

Typical Atterberg limits for various blends of ball clay and fire clay with water are shown in Fig. 6(*a*) and for ball clay and silica flour with water in Fig. 6(*b*). The Atterberg limits were found to be approximately equal to the weighted average of those for each of the constituents based on the per cent by weight of each in the mixture. That is, a line connecting the Atterberg limits of the individual solid constituents (at zero and 100 per cent on the ordinate scale in Fig. 6(*a*) and 6(*b*)) reasonably approximated the data for mixtures of the two. When mixed with clay and water, the silica flour exhibited an "apparent" plasticity as shown by the extrapolated dashed lines in Fig. 6(*b*). This did not occur with sand in the sand-clay-water mixtures.



(*a*) Ball clay-fire clay-water mixtures.
(*b*) Ball clay-silica flour-water mixtures.
Fig. 6.—Atterberg Limits for Various Blends.

Harvard Miniature Compaction:

In order to study the compaction characteristics of the artificial soils, the Harvard miniature compaction apparatus was used. The suggested method (2) for determining moisture-density relationships of soils using this apparatus was

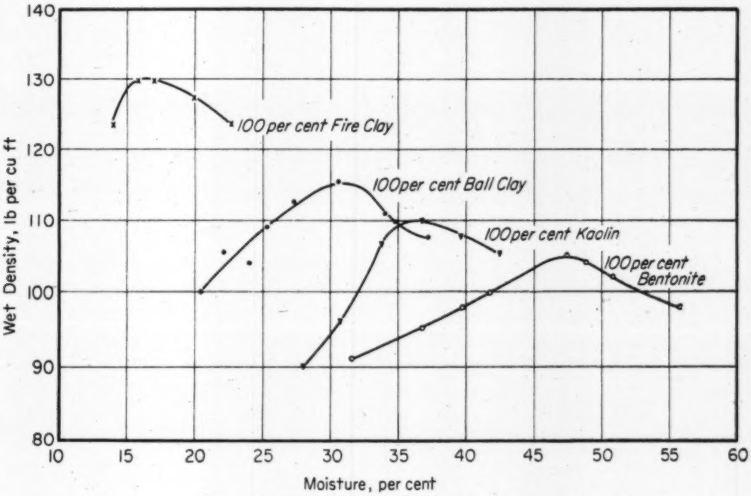


FIG. 7.—Moisture-Density Curves for Various Clays with Water.

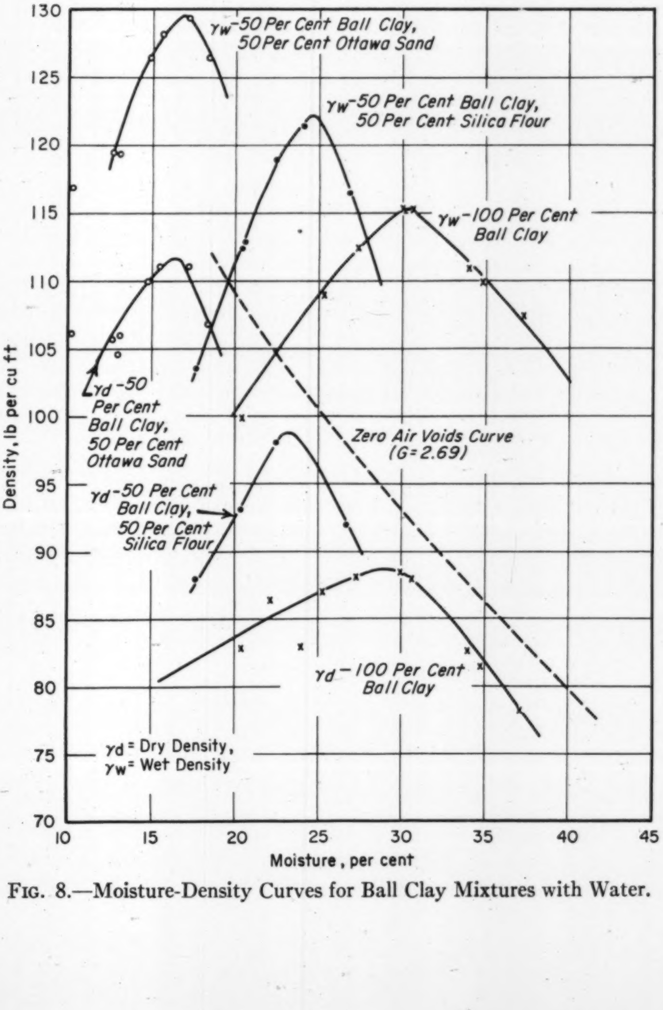


FIG. 8.—Moisture-Density Curves for Ball Clay Mixtures with Water.

followed, except that a different compactive effort was used. It was found that the best procedure for compacting most of the specimens was to use five layers with 10 tamps per layer from the compaction hammer containing a 20-lb spring. For a few of the materials mixed with small percentages of oil or ethylene glycol, especially bentonite, there were some voids and stratification in the sample after molding, indicating the desirability of a greater compactive effort. Rather than change the compactive effort for these few cases, these imperfections were tolerated.

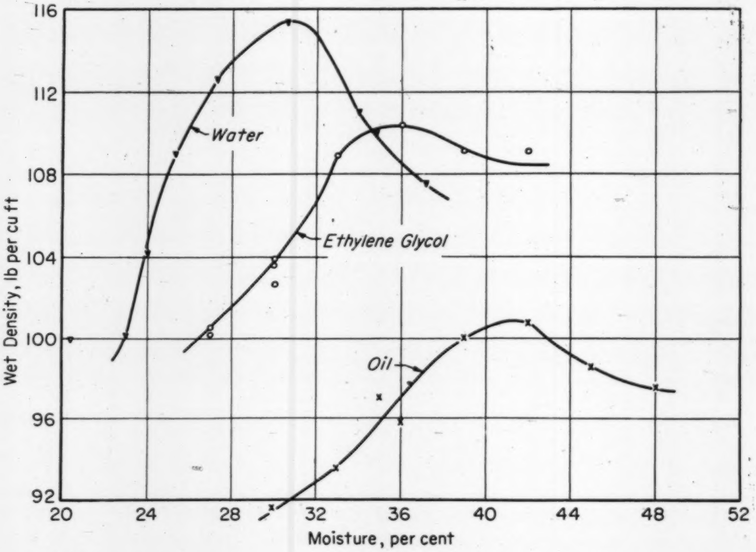


FIG. 9.—Moisture-Density Curves for 100 per cent Ball Clay with Several Liquids.

Typical results of the compaction tests are shown in Figs. 7, 8, and 9. As for the Atterberg limits, when liquids other than water were used they were assigned a specific gravity of 1 in computing the liquid contents. It may be seen in the figures that the optimum "moisture" content is clearly defined for all of the mixtures used. In Fig. 7, the moisture-density curves for each of the four clay minerals mixed with water are compared. In the order of fire clay, ball clay, kaolin, and bentonite, the optimum moisture content increased while the maximum density increased.

Figure 8 shows both the wet-density and dry-density curves for 100 per cent ball clay, 50 per cent ball clay - 50 per cent Ottawa sand, and 50 per cent ball clay - 50 per cent silica flour mixtures with water. This figure shows that in the order of pure clay, clay - silica flour, and clay-sand mixtures, the optimum moisture content decreased while the maximum density increased. The optimum moisture content for the clay-sand mixture based only upon the dry weight of clay present was almost identical to that for the pure clay, indicating that the water was essentially associated with the clay fraction only. In the case of the clay - silica flour mixture, the optimum moisture content was associated with both fractions. In Fig. 9, the effect of the different liquids on the moisture-density

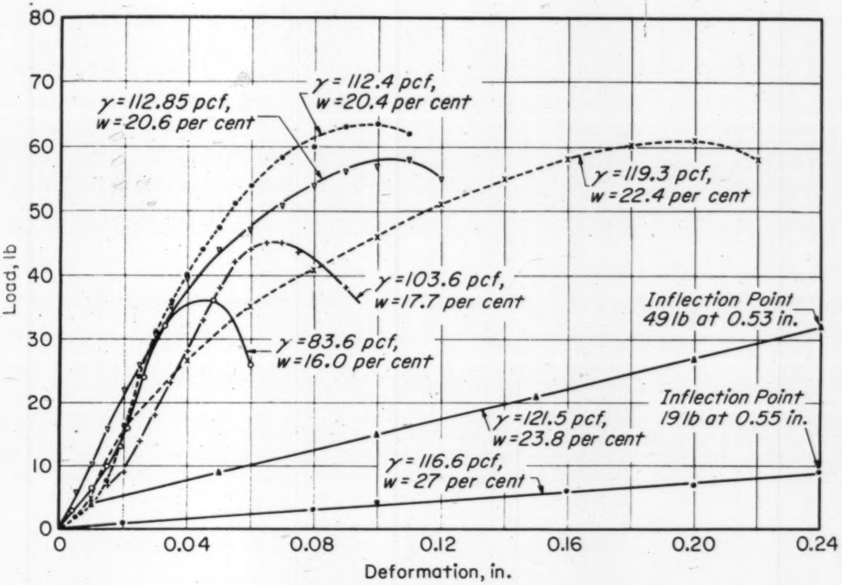


FIG. 10.—Unconfined Compression Load Deformation Curves for 50 per cent Ball Clay-50 per cent Silica Flour with Water in Varying Amounts.

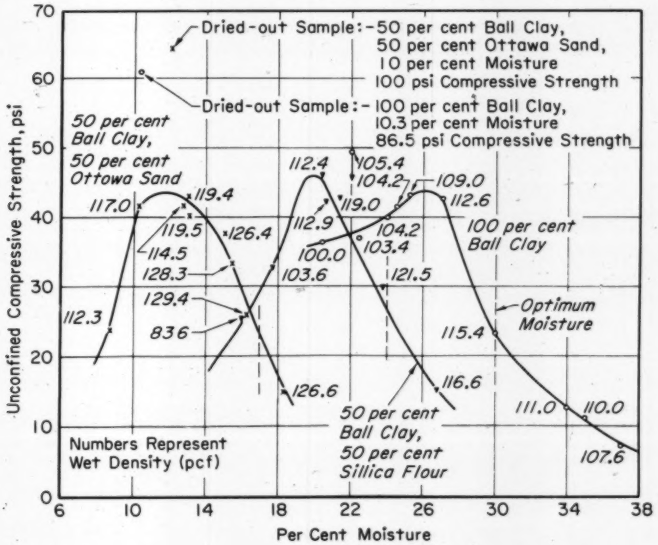


FIG. 11.—Relationship Between Unconfined Compressive Strength and Moisture Content for Several Ball Clay Mixtures with Water.

curves is shown for the unblended ball clay. In the order of water, ethylene glycol, and oil, the optimum liquid content increased while the maximum density decreased.

Unconfined Compression:

The artificial soil specimens for the unconfined compression tests were prepared with the Harvard miniature compaction apparatus with a diameter of 1.31 in. and a height of 2.82 in. The rate of loading was maintained as closely as possible at 0.1 in. per min. Each specimen was loaded until the soil sheared or until the strain reached 20 per cent, whichever occurred first. In all except a few tests at relatively high liquid contents, well de-

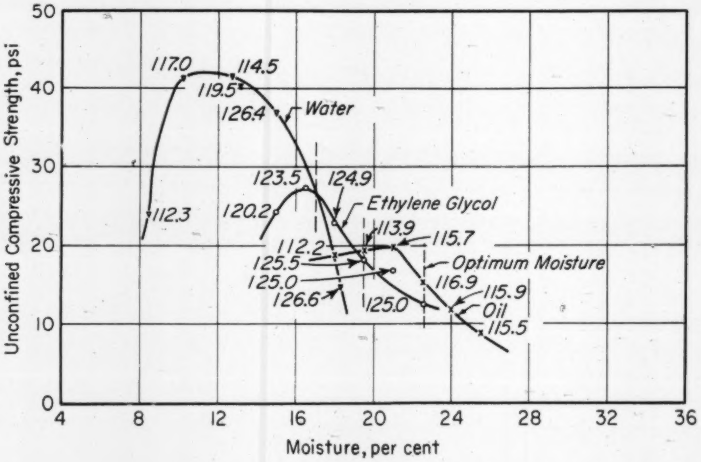


FIG. 12.—Relationship Between Unconfined Compressive Strength and Moisture Content for 50 per cent Ball Clay-50 per cent Ottawa Sand with Several Liquids.

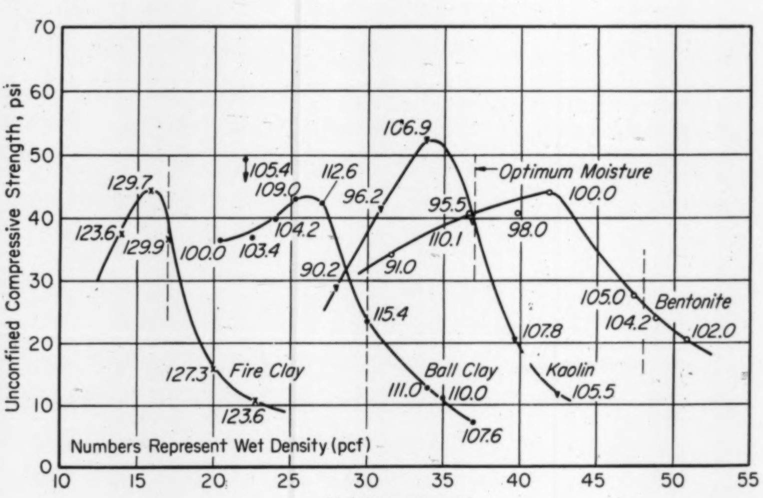


FIG. 13.—Relationship Between Unconfined Compressive Strength and Moisture Content of Unblended Clay Minerals with Water.

fined shear planes developed, especially for the soils mixed with oil.

Unconfined compression load - deformation curves for an artificial soil comprised of 50 per cent ball clay and 50 per cent silica flour with water at a range of moisture contents are shown in Fig. 10. This set of curves is typical of that obtained for each of the other artificial soils tested. As the liquid content increased, the load required for failure increased until it reached a maximum, and then fell off with additional liquid content.

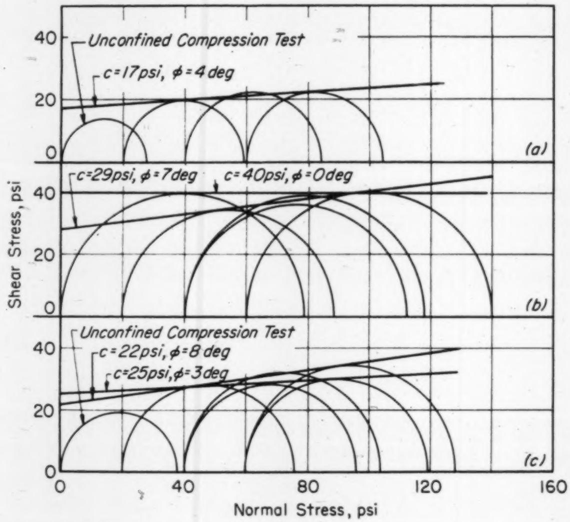
The relationship between the unconfined compressive strength and the per cent liquid content for several artificial soils typical of those tested is shown in Figs. 11, 12, and 13. The figures show that there was a particular liquid content at which the strength was maximum and that this liquid content, in every case, was less than the optimum value based upon the moisture-density tests. The shape of these curves is characteristic of remolded soils where the liquid content during mixing is the same as that at which the sample is to be tested. However, when water is used, the sample may also be prepared at a higher liquid content and the desired amount arrived at by allowing the excess water to evaporate uniformly. In this case, if the soil has plasticity, low final moisture content will generally result in higher strengths than those obtained when drying back is not done. This fact is illustrated by the points above the curves in Fig. 11. This phenomenon has also been observed with natural soils by Baver (3) and others. Apparently it is not possible to distribute small amounts of moisture evenly to all of the clay particles and to orient them for maximum cohesion unless the clay is first saturated and then dried out.

Figure 11 shows that there was only a 10 per cent difference in maximum strength between the pure clay and mixtures of 50 per cent clay with either 50 per cent sand or silica flour. This indicates that the clay was still the predominant factor in determining the properties of mixture. Figure 12 illustrates that the maximum strength that can be obtained for any of the soils with ethylene glycol as the liquid is greater than that which can be obtained with oil, and that with water the strength is greater than with either of the other two liquids. Figure 13 compares the strength curves for the four clays mixed with water. The maximum strength of each was about the same, but occurred at different water contents.

Triaxial Compression:

Unconsolidated, undrained triaxial compression tests were performed on a variety of the artificial soils ranging from 100 per cent clay to 100 per cent sand. The only liquid used was water, and the moisture contents chosen were approximately those at maximum density. Except for the mixtures entirely of sand, the triaxial specimens were molded in the Harvard miniature compaction apparatus in the same manner as for the unconfined compression tests. The sand specimens were prepared in a mold the same size as the Harvard mold by successively dropping a small weight from a given height onto a disk resting on the top of the sand. The weight, drop height, number of drops, and number of layers used were varied to change the density of the compacted specimen.

The axial compression loading rates were held between 0.01 and 0.02 in. per min, and the axial load and deformation of the cylindrical specimens were recorded at periodic intervals of strain throughout the test. Each specimen was tested at several lateral pressures with two identical tests generally being run at each pressure. All specimens containing less than 50 per cent sand showed well-defined shear planes at failure. The specimens containing 50 per cent or more sand merely bulged laterally. The Mohr failure stress circles for these tests have been given in Figs. 14(*a*) to 15(*c*) to show the range of values of cohesion, c, and angle of internal friction, ϕ, which can be obtained.

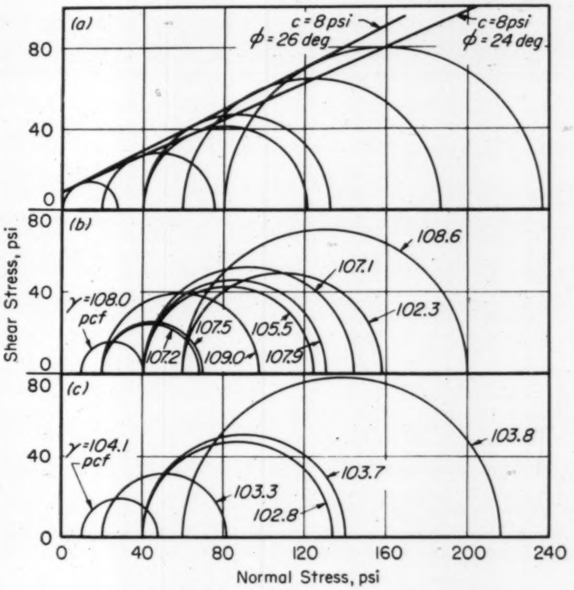


(*a*) 100 per cent ball clay.
(*b*) 75 per cent ball clay and 25 per cent Ottawa sand.
(*c*) 50 per cent ball clay and 50 per cent Ottawa sand.

FIG. 14.—Mohr Failure Circles for Triaxial Tests.

For 100 per cent ball clay specimens an approximate failure envelope fitted to the circles (Fig. 14(*a*)) shows a $\phi = 4$ deg and $c = 17$ psi. In this case the estimate of cohesion from the unconfined compression test was low. For 75 per cent ball clay - 25 per cent Ottawa sand (Fig. 14(*b*)) it is estimated that $c = 29$ to 40 psi and $\phi = 0$ to 7 deg. These values are considerably higher than those for the 100 per cent ball clay specimens. This strength increase was probably due, in part, to the fact that there was a lower liquid content based upon the proportion of clay present (see Fig. 11 for change in strength with change in moisture content). For 50 per cent ball clay - 50 per cent Ottawa sand (Fig. 14(*c*)), it is estimated that $c = 22$ to 25 psi and $\phi = 3$ to 8 deg. For 25 per cent ball clay-75 per cent Ottawa sand (Fig. 15(*a*)) $c = 8$ psi and $\phi = 24$ to 26 deg.

The results for 100 per cent dry Ottawa sand and for 100 per cent dry river sand are shown in Figs. 15(*b*) and 15(*c*), respectively. It is known that the angle of internal friction of sands is a very sensitive function of the density of the material. For this reason it was felt that, rather than drawing an envelope for a number of failure circles, a better estimate of the true ϕ would be obtained by determining the ϕ for each individual circle, assuming $c = 0$, and plotting the results as a function of density. The resulting relationship between density and ϕ is shown in Fig. 16. It is apparent from this figure that ϕ is sensitive to changes in density. It is felt that much of the scatter of data in Fig. 16 is because the density of the specimen was not measured to an accuracy of better than 1 per cent.



(*a*) 25 per cent ball clay and 75 per cent Ottawa sand.
(*b*) Dry Ottawa sand.
(*c*) Dry Chippewa River sand.

FIG. 15.—Mohr Failure Circles for Triaxial Tests.

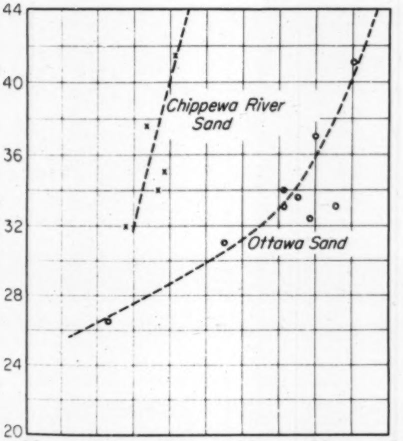


FIG. 16.—Relationship Between Angle of Internal Friction and Density for Dry Sand.

It may also be noted from Fig. 16 that, for a given density, the ϕ for the river sand is higher than that for the Ottawa sand. This is because the Ottawa sand grains are smoother and more regular than those of the river sand (Fig. 1). Since the shear strength of sand changes so significantly with changes in density, in order to obtain reproducible and consistent test results the density must be very carefully controlled and accurately measured.

Mixing and Stability:

During the course of this program, a few studies were made to determine the best way of mixing the artificial soils and to determine the stability of mixtures against change over a period of time.

Most of the artificial soil samples prepared were mixed by hand, using a spatula and a mortar and pestle. Experience with this method showed that it was best to thoroughly mix the solid ingredients first and then to add the liquid. However, hand mixing was limited to small amounts of material. When quantities of more than 1 lb of artificial soil were desired, mixing by hand involved considerable effort and time, and when water was the liquid component, there was uncontrollable loss of moisture during this time due to evaporation.

Mechanical mixing was first attempted with a bench-type, rotary mixer using a mixing bowl. This type of mixer was satisfactory for blending the dry ingredients, but when the liquid was added, the clay lumped into balls, some as large as 2 in. in diameter, and the liquid was not distributed uniformly. A mulling type mixer tried next was found to be more satisfactory. The clay and sand were mixed together, dry, for about 3 min. The liquid was then slowly added through a funnel at the top. Following this, the soil was mixed (about 15 min) until it reached a uniform consistency and color. No large lumps formed with this method.

The effect of changes in water content on some of the properties of soil has been shown in the previous sections. If a sample of soil containing water is exposed to air during the test, water will evaporate from it causing an undesirable change in its properties. Furthermore, if the soil is to be reused, this water must be replaced. In order to overcome this disadvantage when using water, light machine oil and ethylene glycol, non-volatile liquids at normal room temperature, were tried as the liquid constituents.

Although ethylene glycol does not evaporate, mixtures of clay and glycol appear to be unstable over a period of time. It has already been pointed out that when such mixtures are left undisturbed, some of the ethylene glycol comes to the surface. There has not been enough opportunity to find out for what range of liquid content this phenomenon occurs or at what rate. Based upon the results of the unconfined compression tests, it is expected that a change in properties is associated with such a change in liquid content; hence, the ethylene glycol may not have any advantage over water as the liquid ingredient of soil. Oil, on the other hand, appeared to exhibit stability over a period of time. An artificial soil consisting of 50 per cent ball clay and 50 per cent Ottawa sand, with an oil content of 22 per cent based upon the dry weight of the clay and sand, showed no change over a period of six months.

The use of water is not necessarily ruled out when stability over a period of a few days is all that is required. When the size of the artificial soil test specimen is small and when the soil will not be reused, the surfaces exposed to air may frequently be coated with grease or petrolatum to curtail drying. This technique was used in this program in carrying out the unconfined compression tests of the artificial soils containing water. For some of the cases where the soil will be reused, it may be satisfactory to cover the exposed surfaces with a thin, flexible rubber or plastic membrane. In other instances it may be possible to carry out the tests in a closed chamber in which properly controlled humidities can be maintained to prevent water in the soil from evaporating.

SUMMARY AND CONCLUSIONS

The purpose of the study discussed in this paper has been to investigate the properties of artificially prepared soils to determine their value as a research tool for soil mechanics studies. Except for some of the liquids tried, the ingredients of the artificial soils used were all obtained from natural soils. In examining these artificial soils, standard soil identification tests were used so that their soil characteristics could be most easily and meaningfully compared with those of natural soils.

It has been demonstrated that a wide range of such soil properties as plasticity and cohesion, can be achieved with these artificial soils. A similar range of properties could be obtained with a proper selection of natural soils, but not as conveniently, although, if one were willing to sacrifice a little versatility or flexibility, the latter may be the more economical solution when large quantities of soil are required.

Perhaps the most important question that arises in the use of artificial soils is, do these soils behave like real soils. Because of the wide variety of natural soil types that exist it does not appear too difficult to develop ones that do. In this study it was shown that artificial soils could be prepared which would exhibit the same characteristics as natural remolded soils. This certainly does not prove that they can be used as a substitute for natural soils in all respects, but the results do look encouraging.

The extent of reproducibility is basically determined by how similar to the original material are additional materials that can be obtained or prepared. The reproducibility of the artificial soils has been found to be extremely good; that of the natural soils is usually less so unless enough soil is obtained initially to cover all of the tests to be carried out and if it is thoroughly remixed to form a uniform batch.

The major factor in determining controllability is how close one can come to achieving desired values of any soil properties with the materials available. The best chance is with the artificial soils, for which great flexibility has already been demonstrated. Since the properties are a continuously varying function of the proportion of the ingredients present, it is to be expected that any value within the extremes exhibited by the individual constituents can be obtained. A greater variety of choices is usually available with artificial soils than with natural soils. Even with this flexibility, there are probably only a few characteristics that can be independently prescribed to be achieved with a single artificial soil. It would not be practical to try to duplicate all of the properties of a particular soil. Instead, it is best to obtain some of that soil, itself, for the experiments.

Concerning stability, it is known that the properties of a natural soil will vary over a period of time, if only because the water content changes. Operating under a high humidity or covering the soil samples during the test are suggested in this paper as a means of eliminating this problem for a short period of time. Where these methods are not adequate, there is the possibility of using artificial soils mixed with a non-volatile fluid instead of water. Two such liquids were considered in this program, oil and ethylene glycol. It was found that over a period of a few days, some of the glycol gradually separated from the mixture and, hence, this soil was not stable. But the soils containing oil did not change over a period of six months, providing an apparently stable mixture.

Obtaining homogeneity does not pose a problem for either remolded natural soils or artificial soils. It is only necessary to mix the soils thoroughly enough before using it and to set up the specimen properly. Natural undisturbed soils will have to be accepted with whatever degree of homogeneity they possess.

An attempt has been made to set forth and justify the concept of artificial soils as a tool for use in the field of soil mechanics. Much more still needs to be learned about these materials, but it is hoped that the results contained herein will be useful and that further investigations of this subject will have been stimulated.

Acknowledgment:

This study was undertaken as part of the Armour Research Foundation's basic research program. The authors wish to express their appreciation to R. Humphreys and K. W. Miller of the Research and Invention Committee for their many helpful suggestions during the course of this research.

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SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON SOIL DYNAMICS

The impetus to study soil dynamics is due mainly to the increased speeds and loads of present-day vehicles affecting highway subsoils and also to the practice of dynamic precompaction of subsoils. The eight papers in this publication discuss many of the current concepts in the areas of stress-deformation-time relationships and test instrumentation and measurement.

The symposium, sponsored by Subcommittee R-9 on Dynamic Properties of Soils of Committee D-18 on Soils for Engineering Purposes, was held on Monday, June 26, 1961, during the second session of the Sixty-fourth Annual Meeting of the Society at Atlantic City, N. J.

Mr. Rockwell Smith, Association of American Railroads, presided over the session, and Mr. R. K. Bernhard, Rutgers University, was Symposium Chairman.

The papers included in the Symposium are:

Introduction—R. K. Bernhard
Biaxial Stress Fields in Noncohesive Soils Subjected to Vibratory Loads—R. K. Bernhard
Stress-Deformation Relations for Soft Saturated Silt Under Low-Frequency Oscillating Direct-Shear Forces—F. J. Converse
Performance of Embedded Pressure Gages Under Static and Dynamic Loadings—A. J. Durelli and W. F. Riley
Facilities for Dynamic Testing of Soils—G. K. Sinnamon and N. M. Newmark
The Damping Capacity of Some Granular Soils—G. F. Weissmann and R. R. Hart
Testing Procedures for Model Footings and Presentation of TRADEX Site Data—John A. Alai
Dynamic Loading Machine and Results of Preliminary Small-Scale Footing Tests—R. W. Cunny and R. C. Sloan
Bearing Capacities of Dynamically Loaded Footings—S. Shenkman and K. E. McKee
General Discussion

These papers with discussions were issued as *ASTM Special Technical Publication No. 305*, entitled "Symposium on Soil Dynamics."

SUMMARY OF PROCEEDINGS OF THE SESSION ON ROAD AND PAVING MATERIALS AND SYMPOSIUM ON MICROVISCOMETRY

In the volume four general papers are given with discussion that were presented at the thirty-seventh session on June 29. The evaluation of bituminous highway mixtures is an involved subject and encompasses the study of many properties which have significant effect upon performance.

Also included is a Symposium on Microviscometry presented at the thirty-ninth session on June 30 during the Sixty-fourth Annual Meeting of the Society held in Atlantic City, N. J. The sliding plate microviscometer appears to have considerable promise as a suitable type of apparatus for the measurement of this very significant property.

Mr. J. O. Izatt, Shell Oil Co., Inc., presided over the thirty-seventh session and Mr. A. B. Cornthwaite, The Asphalt Institute, presided over the thirty-ninth session.

Papers on Road and Paving Materials

A Laboratory-Field Study of Hot Asphaltic Concrete Wearing Course Mixtures—Joseph F. Goode and Elliott P. Owings

The Significance of Variation in Asphalt Content of Paving Mixtures—J. Hode Keyser and N. G. Gaudette

Immediate Core Volume by Presaturation—L. E. Santucci and R. J. Schmidt

Air Permeabilities of Asphalt Concrete—T. C. Hein and R. J. Schmidt

Measurement of Asphalt Viscosity with a Vacuum Capillary Viscometer—J. J. Heithaus

The papers comprising the Symposium are:

Symposium on Microviscometry

Introduction—C. L. Monismith

Measurement of the Consistency of Paving Cements at 140 F with the Sliding Plate Microviscometer—D. F. Fink and R. L. Griffin

The Precision of Measurements with the Sliding Plate Microviscometer—D. F. Fink and J. J. Heithaus

The Rheology of Asphalt-Filler Systems as Shown by the Microviscometer—R. S. Winniford

Effects of Viscosity in Bituminous Construction—Verdi Adam

Changes in Asphalt Viscosities During the Thin Film Oven and Microfilm Durability Tests—Woodrow J. Halstead and Joseph A. Zenewitz

These papers with discussion are issued as *Special Technical Publication No. 309* entitled "Papers on Road and Paving Materials and Symposium on Microviscometry."

THE EFFECT OF SECTION SIZE ON THE NOTCH STRENGTH AND FRACTURE DEVELOPMENT IN SELECTED STRUCTURAL METALS*

By E. P. Klier[1] and Volker Weiss[2]

Synopsis

Cylindrical sharp-notch tension specimens of gross section sizes up to 2 in. in diameter have been tested at room temperature. Steel, titanium, and aluminum materials in both strengthened and softened structural conditions have been examined to establish the effect of test specimen size on the notch properties.

The changes in notch geometry at the point of fracture initiation have been studied as function of specimen size for unalloyed titanium. Limited test data are offered to show that notch geometry changes must play an important role in the phenomenon of size effect.

Crack development in the cylindrical notched specimens is discussed at length. It is shown that the notched specimens separate first near the base of the sharp notch but that final separation for concentrically loaded specimens results ultimately from the nucleation of an internal fracture probably due to ductility exhaustion which propagates to separation of the specimen.

During the past several years the growing need for increasingly high-strength notch-tough thin-section materials for rocket and space vehicle applications has been emphasized. Paralleling this development has been an increasing need for materials with high strength and notch toughness in heavy sections. It would seem that the fracture problems in both light and heavy sections are closely related and ultimately may be encompassed within the same theoretical framework, but this can be practically meaningful only if the model laws governing crack propagation are soundly developed. This means that detailed descriptions of the conditions under which cracks form and grow—not at present available—must be developed. The purpose of the present work is to offer a preliminary study of the changes in notch properties and crack development that occur in selected structural materials in low- and high-strength conditions as the section size is changed.

In all, a heat-treated steel, alloyed and unalloyed titanium, and low- and high-strength aluminum materials have been examined in the cylindrical notch tension test at room temperature, for sizes variously ranging up to diameters of 2 in. The effects of heat-treating cycles were studied for the steel and the fracture development was examined for all conditions.

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* Presented at the Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

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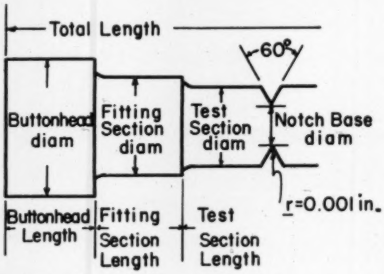
[2] Associate Professor, Chemical and Metallurgical Engineering, Syracuse University, Syracuse, N. Y.

TABLE I.—COMPOSITIONS IN PER CENT OF MATERIALS INVESTIGATED.

Material	Carbon	Manganese	Phosphorus	Sulfur	Silicon	Chromium	Nickel	Iron	Molybdenum	Nitrogen	Oxygen	Hydrogen
4340	0.43	0.73	0.012	0.022	0.26	0.78	1.9	balance	0.24	...	...	...
Titanium 150-A	0.04	w[a]	...	...	w[a]	2.75	...	0.35	...	0.078	0.26 0.24	0.013 0.011
Titanium, unalloyed	0.07	w[a]	...	...	vw[a]	...	...	...	...	0.012	0.10 0.11	0.0017 0.0017

Material	Manganese	Chromium	Iron	Iron + Silicon	Copper	Magnesium	Zinc	Titanium	Aluminum	Other Elements
Aluminum 7075-T6 (nominal composition)	0.30	0.18 0.40	0.7	...	1.2 2.0	2.1 2.9	5.1 6.1	0.20	balance	0.15
Aluminum 1100-0 (nominal composition)	0.05	...	...	1.0	0.20	...	0.10	...	99.0 min	0.15

[a] Spectrographic analysis: 0.01 per cent $< w <$ 0.1 per cent.
0.001 per cent $< vw <$ 0.01 per cent.



Specimen dimensions in inches for 4340 steel:				
Test section diameter (plotted diam)	0.300	0.500	1.100	1.500
Notch base diameter	0.212	0.353	0.800	1.100
Test section length	1.00	1.75	1.5	1.5
Buttonhead length	0.25	0.625	1.5	1.5
Fitting section length	...	...	0.75	0.75
Fitting section diameter	...	...	1.99	1.99
Buttonhead diameter	0.5	0.75	2.99	2.99
Total length	1.5	3.0	6.0	6.0

Specimen dimensions in inches for aluminum and titanium materials:					
Test section diameter (plotted diam)	0.300	0.500	0.900	1.400	2.000
Notch base diameter	0.212	0.353	0.635	0.990	1.413
Test section length	1.00	1.75	1.5	1.5	3.0
Buttonhead length	0.25	0.625	1.5	1.5	1.5
Fitting section length	...	...	0.75	0.75	...
Fitting section diameter	...	...	1.99	1.99	...
Buttonhead diameter	0.5	0.75	2.99	2.99	2.99
Total length	1.5	3.0	6.0	6.0	6.0

FIG. 1.—Notch Tension Specimen Dimensions.

EXPERIMENTAL PROCEDURE

Materials:

A 4340 steel heat treated to selected strength levels, two aluminum alloys, and two titanium alloys were tested. The compositions of these materials are given in Table I.

All materials were supplied as forged bar 3 in. or slightly larger in diameter.

Steel Heat Treatments:

In the hardening of the 4340 steel three austenitizing treatments were differentiated as follows:

1. "Conventional heat treatment" consisting of austenitizing at 1550 F for 4 hr followed by oil quenching. Tempering followed immediately. This treatment is designated in the graphs as 1550 (4) OQ.

2. "Two-step treatment" consisting of austenitizing at 1550 F for 4 hr, followed by furnace cooling to 1350 F, holding for 1 hr, and oil quenching. Tempering followed immediately. This treatment is designated as 1550 (4) FC 1350 (1) OQ.

3. The "Lockheed treatment" as specified in "Lockheed Process Bulletin 220-M" (**1**),[3] consisting of austenitizing at 1625 F for 2 hr, normalizing, reheating to 1525 F for 2 hr and oil quenching. (The retained austenite stabilization treatments specified were not used.) This treatment is designated as 1625 (2) AC 1525 (2) OQ.

It was expected that tempering time would influence the notch strength, and this factor was investigated for times of 1, 4 and 12 hr, at the temperatures entered in the figures.

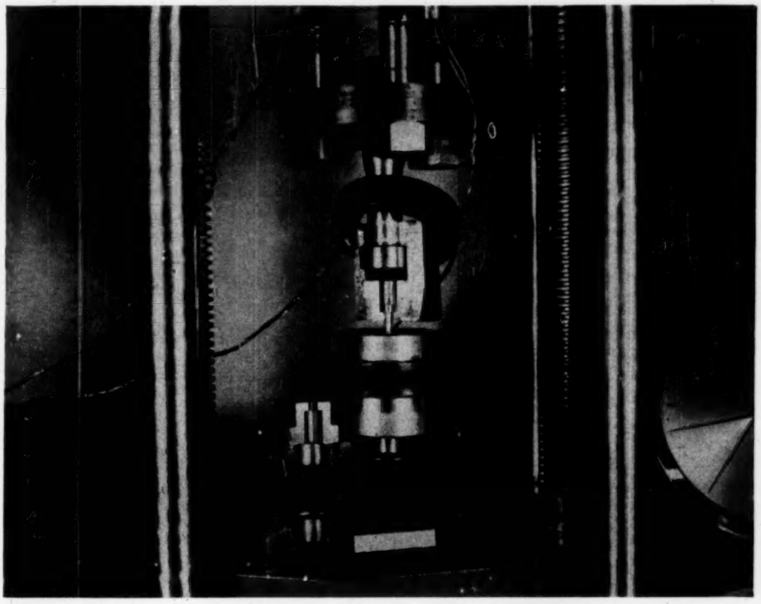


FIG. 2.—Concentric Test Fixture with Adaptors.

Notch Test Specimens:

The test specimens were oriented in the rolling direction. For all specimen diameters the notch was the 50 per cent 60-deg V notch with a maximum notch root radius of 0.001 in. The steel specimens were heat treated in a rough machined condition and were finish machined after heat treatment.

Several sizes of notch buttonhead tension specimens were used (Fig. 1). For steels the testing capacity available did not permit fracturing of ductile 2-in. diameter specimens.

Tension Testing Procedure:

Specimens were loaded in the adaptors

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

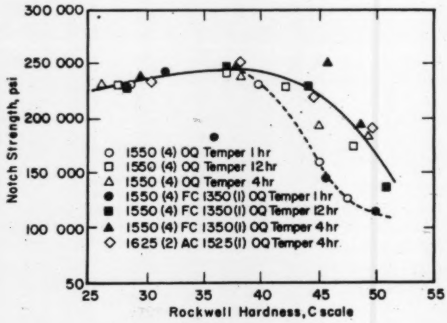
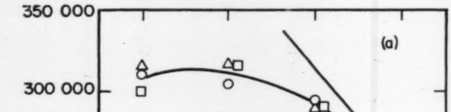


FIG. 3.—Notch Strength Versus Hardness with Tempering Time as Parameter for the Indicated Austenitizing Treatments; 4340 Steel, 1.1-in. Diameter Notch Specimens.



(*a*) Notch strength versus tempering temperature with specimen diameter as parameter.

(*b*) Fracture ratio versus tempering temperature for 1.1-in. diameter notch tension specimens.

FIG. 4.—Notch Properties of 4340 Steel Versus Tempering Temperature.

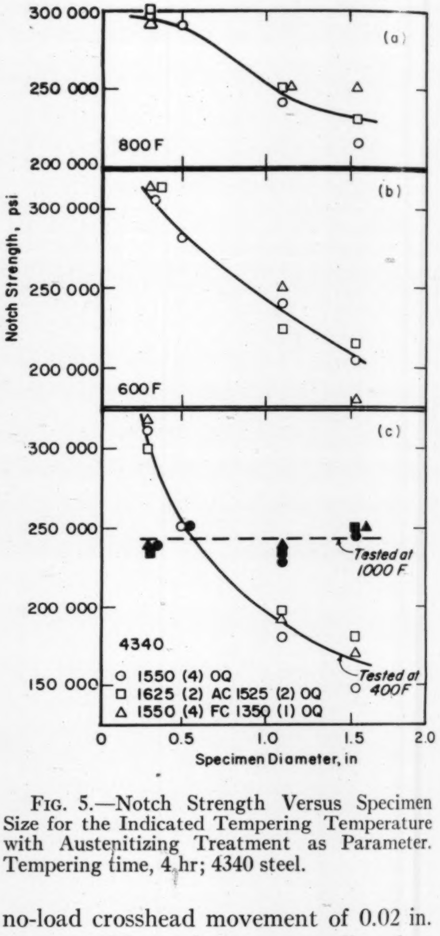


FIG. 5.—Notch Strength Versus Specimen Size for the Indicated Tempering Temperature with Austenitizing Treatment as Parameter. Tempering time, 4 hr; 4340 steel.

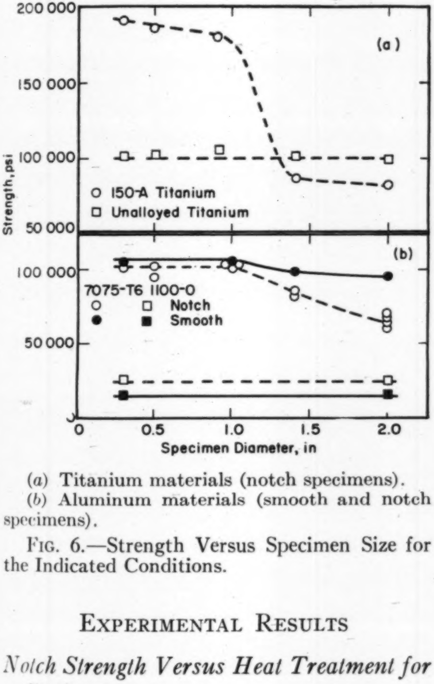
illustrated in Fig. 2 at room temperature at a loading rate corresponding to a no-load crosshead movement of 0.02 in. per sec. All specimens were aligned at the start of the test to be equidistant from the columns in the testing machine.

Fracture Examination:

Fracture surfaces were examined and steel specimen fractures were photographed. (A report of these fractures has been prepared (2) and in limited measure circulated. However, the report is not generally available.) A limited number of these fracture photographs are presented in the present paper. All fracture surfaces were examined for shatter cracks, an example of which is given in Fig. 16(*b*).

Hardness Surveys:

Hardness measurements were completed on the test section for all steel specimens as a check of properties variations through the sections. The average of the measurements is entered in the figures. An example of the indentation array taken is given in Fig. 16(*b*).



(*a*) Titanium materials (notch specimens).

(*b*) Aluminum materials (smooth and notch specimens).

FIG. 6.—Strength Versus Specimen Size for the Indicated Conditions.

EXPERIMENTAL RESULTS

Notch Strength Versus Heat Treatment for Steels:

The notch properties in a heat-treated steel vary with changes in the heat treatment. In the present work three austenitizing treatments and three tempered conditions were evaluated. The results are presented in Fig. 3. Here it is indicated that for strength conditions corresponding to about Rockwell 40, C scale, or less the method of heat treatment is of secondary importance. At higher hardnesses, however, a most important factor is tempering time. For the steel investigated at 1.1 in. diameter maximum properties are obtained for a tempering time of 4 hr or more. A tempering time of 1 hr produces definitely inferior notch properties.

Notch Strength Versus Test Specimen Size:

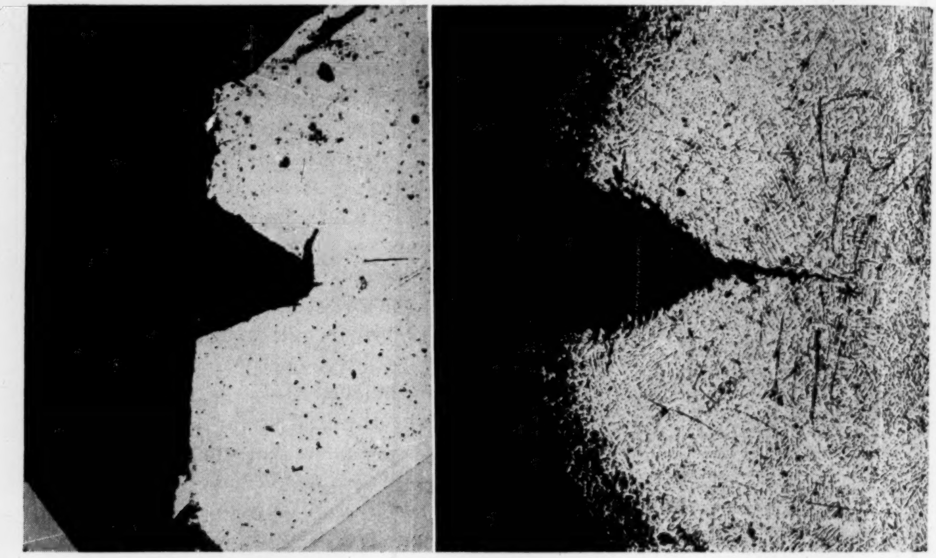
The effect of test section size on the notch strength in steel is summarized in Fig. 4. In brief, for low-strength conditions, corresponding to a low hardness, the notch strength does not vary with section size. As the strength level is raised, strength conditions are eventually attained at which notch sensitivity sets in with increase in section size. These changes are perhaps best illustrated for the data on steel in Fig. 5.

At the lowest hardness level examined, the notch strength remains constant as the section is enlarged. As the tensile strength level rises, the notch strength initially is raised proportionately for the small specimens and remains essentially constant to intermediate sizes and then begins to drop with further increase in section size (800 F temper). As the strength level is further increased, the notch strength for small specimens does not further increase while the size effect phenomenon sets in at increasingly smaller specimen diameters. Because of this a strength condition which yields maximum notch strength at the smallest size may give the lowest notch strength at the largest test size. Parallel trends are clearly indicated for titanium and aluminum materials (Fig. 6).

For the aluminum materials, large sized tension specimens were also tested. It is evident that for the high-strength alloy merely a change in the tension specimen size is sufficient to cause a reduction in the strength properties. Similar reductions in tensile strength with increase in specimen size have also been reported for high-strength steel conditions (3).

Notch Geometry Changes in Circumferentially Notched Cylindrical Bars:

Practical difficulties limit the notch dimensions that can be machined into cylindrical test bars. However, notch root radii of the order of 0.0001 in. have been formed in a limited number of small steel specimens (4). Experimental results obtained with such notches compared favorably with those obtained with notches of 0.001-in. root radii. From this it was concluded that the notch root radius of 0.001 in. largely produces the most severe notch effect and sharper notches develop no new results. Since even in the small cylindrical specimens notch dimensions of 60-deg flank angle, root radius 0.001 in., and 50 per cent removal of the section have a theoretical elastic stress concentration factor of 10, it is evident that in the presence of even slight ductility at the notch base the overloading due to the notch will quickly be redistributed or the section must part. If the specimens are geometrically scaled to have the same theoretical stress concentration, and if the fracture phenomenon scales in the same way, specimen behavior to the initiation of failure should be the same for all specimens. The fact that contrary results are regularly observed (5) constitutes the phenomenon of size effect. Explanation of this effect may be advanced as resulting from a critical balance between elastic and plastic strain energy as proposed by Irwin (6). The effect may also be explained as due to modification of notch geometry due to differences in plasticized zone development through the set of different sized specimens. To examine this question a set of ductile titanium specimens of different sizes were pulled to initiate cracking at the root of the



(*a*) 0.3-in. diameter specimen. (*b*) 2.0-in. diameter specimen.

FIG. 7.—Notch Root Geometries in the Smallest and Largest Size Titanium Specimens at Approximately Maximum Load (×16).

Initial notch radii were equal to 0.001 in.

notch. The specimens were not broken but were removed from the test machine and sectioned. The notch geometries were established by sectioning through the centerline of the specimen as is evident from Fig. 7. Here it is seen that in the small specimen the notch root radius has been significantly enlarged without the development of a crack. In the larger specimens notch radii have not been enlarged, but rather cracks have developed and these are longer, the larger the test specimen. From this it follows that notch action in the small specimens, even for the most sharply notched conditions that have been ground and machined, is not as severe as the conditions that obtain in large specimens.[4] However, if such small specimens can be notched

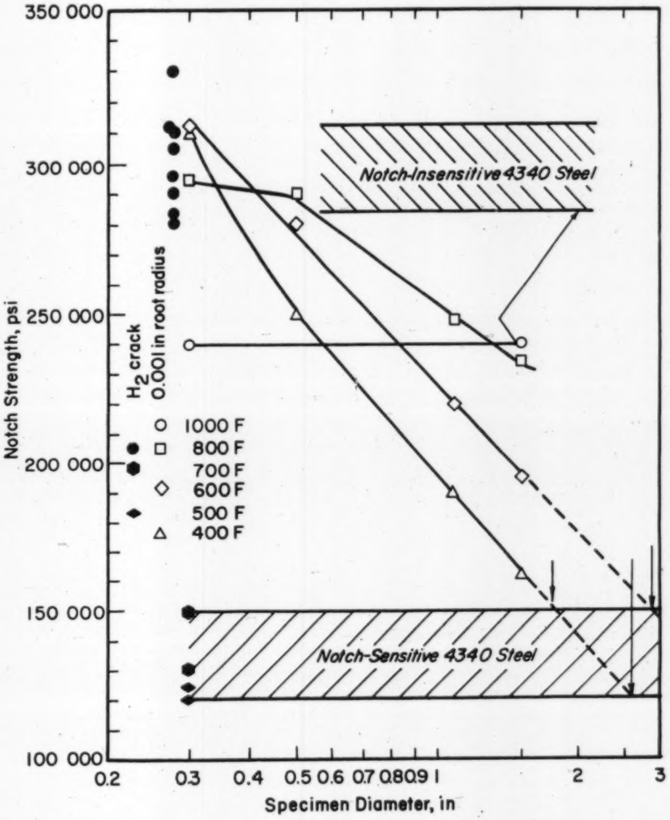


Fig. 8.—Notch Strength Versus Log Specimen Diameter Compared with Limiting Notch Strengths Measured for Hydrogen-Cracked Small Specimens of 4340 Steel.

Note.—On the basis of the data presented in Klier, Muvdi, and Sachs (**12**) it is assumed that in the presence of a hydrogen crack the size effect can be eliminated if the data are properly reduced. The two banded zones labeled "Notch-Insensitive" and "Notch-Sensitive 4340 Steel" were drawn on the basis of this assumption.

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[4] It is evident that the phenomena here considered are precrack phenomena and are not contingent on a fracture mechanics hypothesis in any form. The effect observed suggests that mechanical similarity requirements must also pertain to scaling of the microconstituents in the respective sections, which proposal has been made elsewhere (**7**).

without the introduction of the large plasticized zones which in the machined notch specimens lead to notch geometry changes, notch restraint in the small specimens potentially may be increased and attain the same order of magnitude as that in the large specimens. Two possible methods exist for producing the ultra-sharp notches required—namely, through combined chemical-mechanical means as, for example, exist in hydrogen embrittlement; and, secondly, mechanically, through the controlled development of a fatigue crack. The limited data available for the comparison of notch action arising from these two sources indicate that for both crack forms the same notch properties are observed (8,9).

In an earlier publication (12) the results of a preliminary study of hydrogen embrittlement as modified by size effect are reported. In this study it was pointed out that for certain heat-treated conditions the introduction of the hydrogen crack led to no reduction of the instantaneous notch strength. These conditions have been considered as notch-tough or notch-insensitive. At higher strength levels for the 4340 steel studied the hydrogen crack led to reduced instantaneous notch strength. This has been taken as evidence of notch sensitivity. From the results of this and related studies it has been assumed that the upper limit of notch strength for a notch-tough condition is ≈ 300,000 psi while the lower limit for a notch-sensitive condition is ≈ 125,000 to 150,000 psi. The value of 300,000 psi for the notch-tough condition can be measured in practice only if the yield strength of the steel is sufficiently high or if geometrical restraint can be developed to a suitable degree. The exact values of the two projected notch strength conditions cannot be established from the measurements that have been completed to this time, but the results of the hydrogen-embrittlement size effects and correlated studies suggest that both for the notch-tough and notch-sensitive 4340 steel conditions the phenomenon of size effect may enter only accidentally. It should then be possible to design experiments in which the results would be unaffected by size effect. The hydrogen-cracked specimens are proposed as specimens for which the size effect factor has been eliminated and the barred zones are entered in Fig. 8 on this basis. The general meaning of this figure would then be as follows:[5]

1. For low yield strength, notch-tough conditions, a notch strength of less than 300,000 psi would be expected and this should be independent of size of specimen, if the geometry is retained.

2. If the geometry is altered, the notch strength should change, and if the geometry is changed to increase restraint, the notch strength should increase. For 4340 steel tempered at 1000 F this is indicated as developing for the notch strength measured for the instantaneous notch area.

3. For the 4340 steel a notch strength of 300,000 psi in the small specimens with machined notches is accompanied by a definite size effect.

4. The specimen diameter interval in which the factor size effect is measured may be determined by extrapolation of the notch strength - diameter curves. For machined notches this leads to minimum specimen diameters of the order of 2 in. for minimum notch strength measurements, and as the tempering temperature is allowed to increase this minimum diameter rapidly increases.

However, the small-specimen data are

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[5] The 4340 steel used in the hydrogen embrittlement study contained 0.38 per cent carbon; the 4340 steel used in the size effect study contained 0.43 per cent carbon. This carbon content differential is believed to be responsible for the somewhat different notch sensitivities indicated for the two steels.

not at this time considered other than as illustrating trends and are not offered as definitive. Nonetheless, these data demonstrate that small specimens can be so prepared that the notch action effect is more severe than that measured for the largest sized specimens used in the present investigation. Work has been projected to test large sized specimens, and it is evident that the possible correlation of such tests with the small-specimen tests here described would prove extremely valuable.

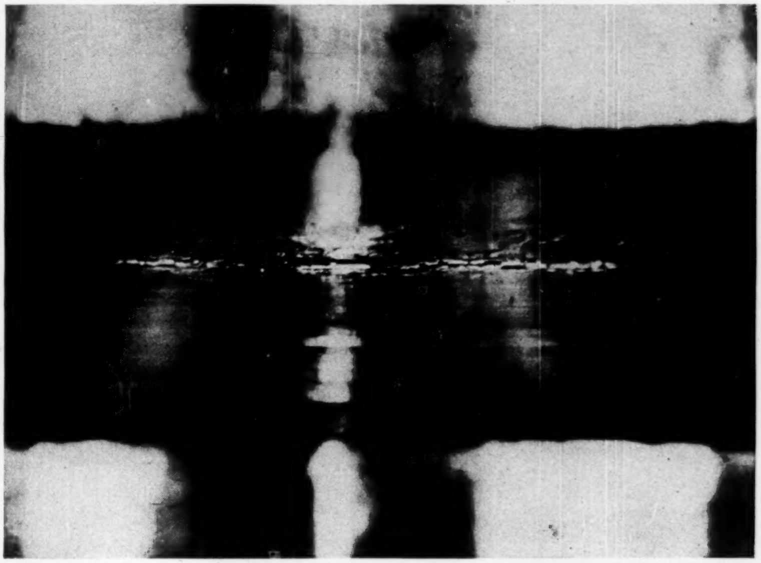

FIG. 9.—Circumferential Crack Development in a Notch-Tough Steel Specimen Loaded at Room Temperature; Specimen Diameter 1.1 in.

Crack Development in Circumferentially Notched Bars:

The development of the crack in circumferentially notched bars has been described at length for conditions of hydrogen embrittlement. The hydrogen crack after initiation may develop externally or internally, and through hydrogen-containing or hydrogen-barren materials (**10,11,12**). In the absence of hydrogen embrittlement, relatively few studies have been directed at determining the course of crack propagation away from the notch base.

Earlier it has been pointed out that for sharp notches all cracking for circumferentially notched specimens begins in the immediate vicinity of the notch base. This is illustrated in some measure in Fig. 9. The exact development of this crack is extremely difficult to establish but it can be inferred that the crack, while probably initiated at a localized point in the vicinity of the notch base, develops at a rapid rate around the notch. For a brittle material the possibility exists that propagation of the fracture through the specimen might take place instead of propagation of the crack around the notch, and this mechanism has been proposed recently by Irwin (**13**) for high-strength steel test specimens of the type under study. It would appear, however, that the mechanism proposed by Irwin is obtained only when significant eccentricity of load application arises. For concentric load application the mechanism of crack development is believed to be otherwise.

In ductile metal, the notch is not fully effective in restraining plastic flow. In an earlier section it has been proposed that such plastic flow leads to specimen geometry changes which significantly contribute to the phenomenon of size effect. This behavior is most evident for small specimens and has been described at length for titanium (14). However, even for the largest specimens investigated in this study, the low-strength aluminum deforms outside the notch (Fig. 10). For this specimen the crack was initiated at the notch base and then moved inward. Late in the separating process, it appears that a crack developed internally and moved to meet the advancing circumferential crack. A very small segment of the section remained joined just prior to final separation. As the strength of the material increases, the development of the crack progressively changes, but vestigial evidence of the crack propagation can clearly be correlated with the fracture process just described for the soft aluminum bar. In Figs. 11(*a*) to (*d*) a progressive series of fracture facets in high-strength steel test specimens is presented.

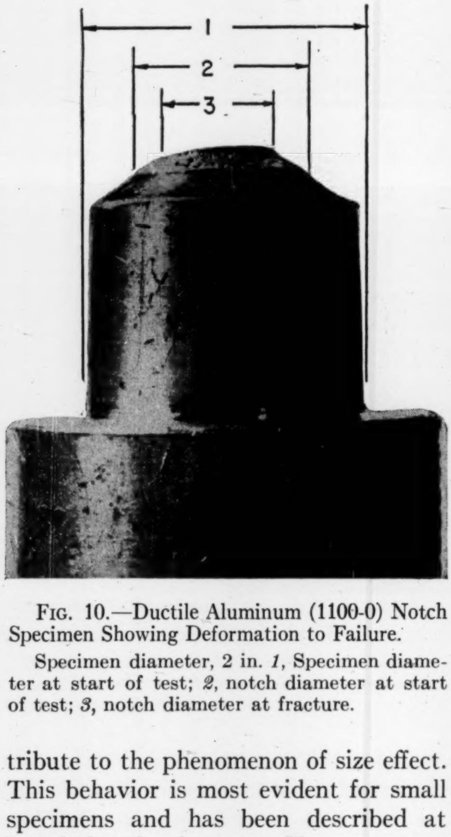

FIG. 10.—Ductile Aluminum (1100-0) Notch Specimen Showing Deformation to Failure.

Specimen diameter, 2 in. *1*, Specimen diameter at start of test; *2*, notch diameter at start of test; *3*, notch diameter at fracture.

In the specimens examined there is evidence of the circumferential crack at the notch base.[6] However, evidence of this circumferential crack in steel specimens is difficult to register satisfactorily in a photograph. One of the best examples is presented in Fig. 12. In the oriented sketch the extent of crack penetration from the notch base is indicated. It is readily evident that the crack developed to a much greater extent at the top of the macrograph, while the limits of this penetration are relatively easy to establish.

Opposite the point of maximum penetration of the surface crack the internal crack nucleus is identified. The position of this crack nucleus is believed to be determined by the ductility of the steel and moves increasingly toward the center of the fracture as the ductility increases. This is indicated in the figures. The distance of this fracture nucleus from the notch base divided by the half-diameter of the fracture is called the fracture ratio and this quantity has been plotted in Fig. 4.

It is proposed, then, that the section is separated by the development of internal cracks which propagate out as the circumferential crack propagates in. It is believed that the fracture facets under discussion developed strictly parallel to

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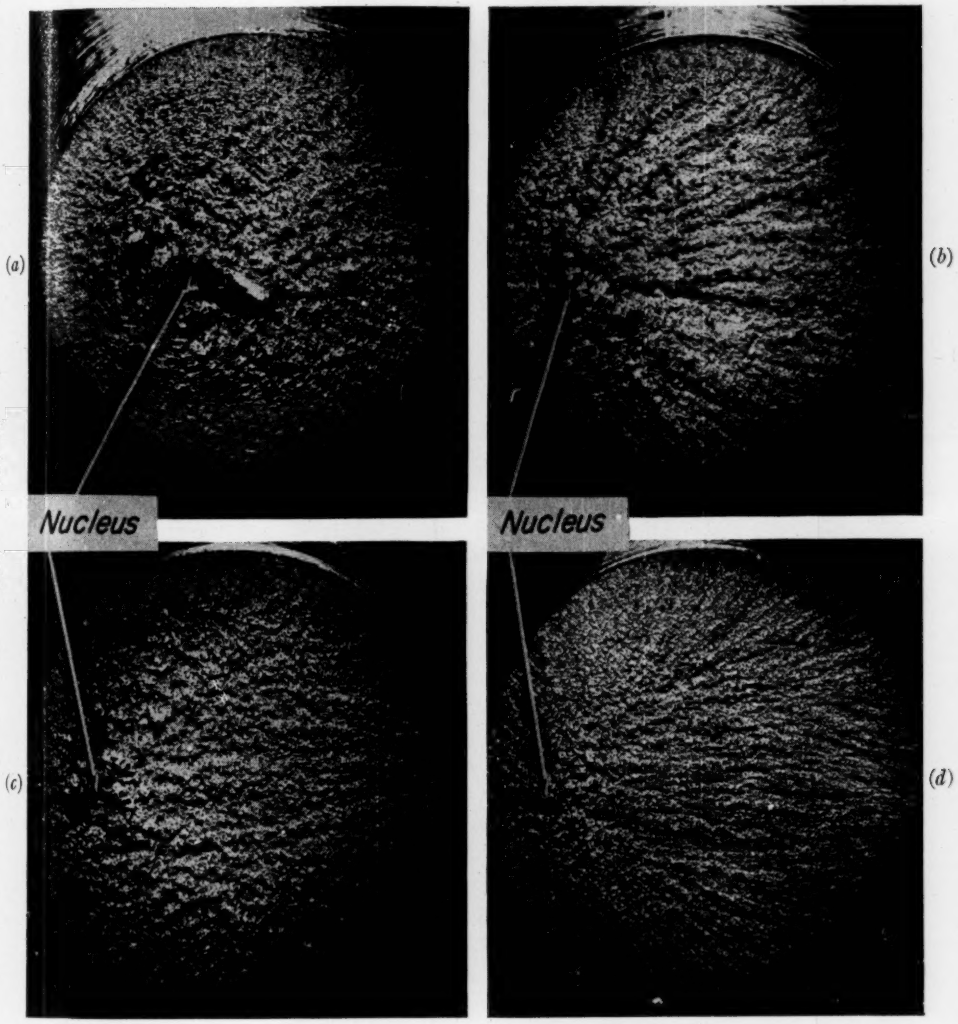
[6] For the softer tempered conditions this peripheral crack development was regularly observed (see Fig. 9).



(*a*) 1550 (4) FC 1350 (1) OQ. Temper: 1000 (1); notch strength: 246,000 psi; Rockwell hardness, C scale: 32.1.

(*b*) 1625 (2) AC 1525 (2) OQ. Temper: 800 (4); notch strength: 254,000 psi; Rockwell hardness, C scale: 37.3.

(*c*) 1625 (2) AC 1525 (2) OQ. Temper: 600 (4); notch strength: 219,400 psi; Rockwell hardness, C scale: 44.5.

(*d*) 1600 (4) 1500 (2) OQ. Temper: 400 (4); notch strength: 191,400 psi; Rockwell hardness, C scale: 49.5.

Fig. 11.—Fracture Surfaces of 4340 Steel Following Various Heat Treatments (Specimen Diameter 1.1 in.).

that observed for the aluminum test bar. For this condition of fracture to be realized, however, a minimum ductility must exist. In addition to these ordinary fracture types somewhat different fracture patterns may develop.

For example, it has been stated that the circumferential crack develops at a rapid rate about the base of the notch, probably from a local surface nucleus. This takes place because radial propagation of a crack from a surface location tends to unload the crack front. Under this condition the only way in which the crack front can be progressively loaded as it moves away from the notch base is for the test specimen to be loaded in bending. If this is prevented, the load will be shifted progressively or abruptly from the location of the surface fracture nucleus to the opposite side of the bar. If the load shift is made progressively, a screw shift in the load develops and the crack moves around the notch base. This may possibly characterize crack development for the low-strength conditions in the steels. If for any reason the neutral axis of the stress system moves directly away from the fracture nucleus,[7] an oscillatory shift in load and crack development may develop. An example of crack development in this manner is illustrated in Fig. 13. In this example, circumferential crack development took

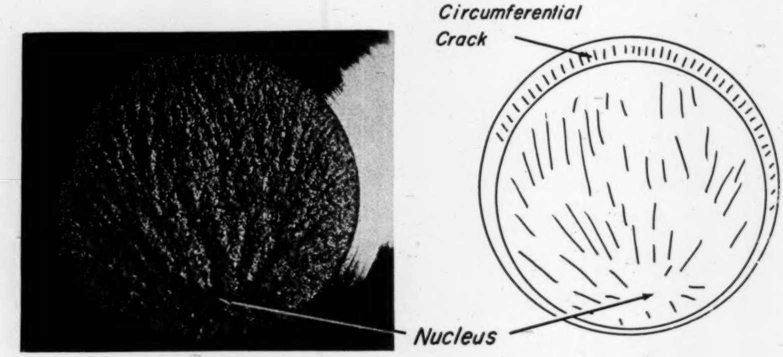


FIG. 12.—Fracture Surface Developed in 4340 Steel Notch Specimen.

The limits of surface crack penetration prior to failure from the indicated internal fracture nucleus is indicated in the sketch. Heat treatment: 1550 (4) OQ; 800 (1). Specimen diameter 1.1 in.

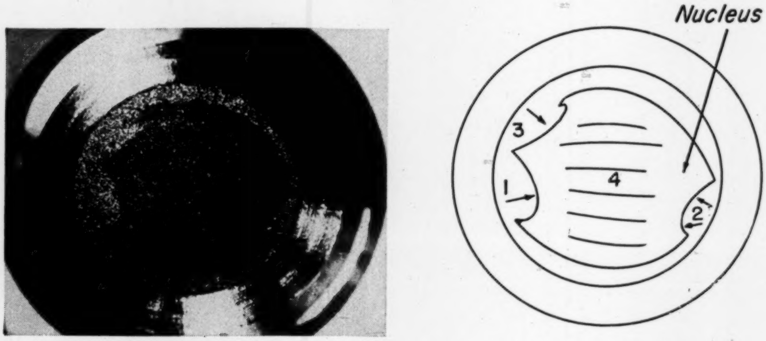


FIG. 13.—Fracture Surface of Unalloyed Titanium Notch Specimen Illustrating Complex Surface Crack Development Prior to Final Separation of the Section.

A possible sequence for crack development is indicated in the sketch. Specimen diameter 0.9 in.

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[7] Peripheral crack development could be suppressed if the fracture nucleus were bounded by material of higher ductility, as for the example in Fig. 13.

place and was followed by what appears to have been three oscillations in load during which the specimen remained intact. On the fourth oscillation the specimen broke. This mode of fracture development is believed to characterize breaking in the high-strength steel materials, with failure following the first oscillation in load.

In the absence of bending the load shift described above must always take place, and the crack propagation into the specimen can take place only if the notch properties change as the crack propagates radially in. (For certain material conditions this change in notch properties seems to be possible.) The actual course of crack propagation beyond the development of the initial circumferential crack at this time can best be established by examination of the fracture surface.

To assist in the evaluation of fracture surfaces it is necessary to introduce only the concept of fan pattern development of the fracture and to emphasize that this pattern is characteristic (15). The fracture nucleus is at the origin of the fan. Cracks that are surface nucleated but which run internally have been discussed for conditions of hydrogen embrittlement (12). A fracture pattern for a sharp-notched specimen which by the criterion used failed by the internal propagation of a surface nucleated crack is given in Fig. 14. Fracture development as illustrated here faithfully portrays the fracture pattern required for the fracture mechanism advanced by Irwin (12).[8] There are definite similarities between fracture facets originating and fully developing from the surface and those observed in the present work. However, the fan patterns are inverted. For this reason the fractures in the present work are proposed to develop from the location which has been described as the fracture nucleus, located at an internal fracture site.[9]

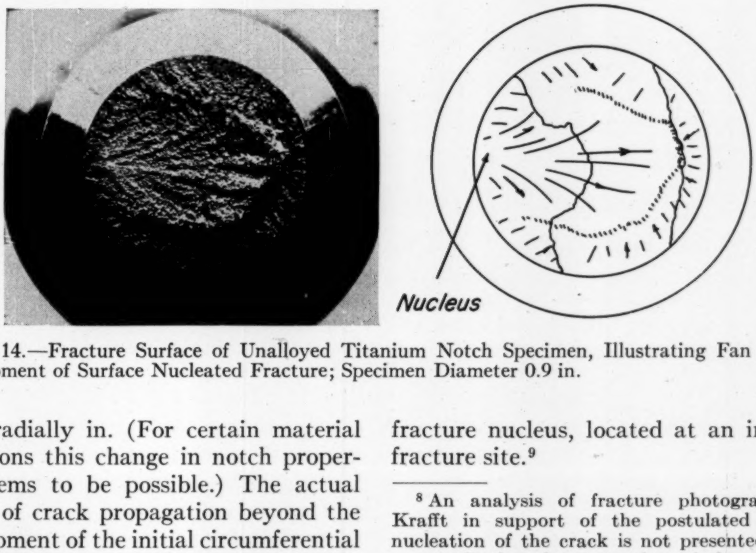

Fig. 14.—Fracture Surface of Unalloyed Titanium Notch Specimen, Illustrating Fan Pattern Development of Surface Nucleated Fracture; Specimen Diameter 0.9 in.

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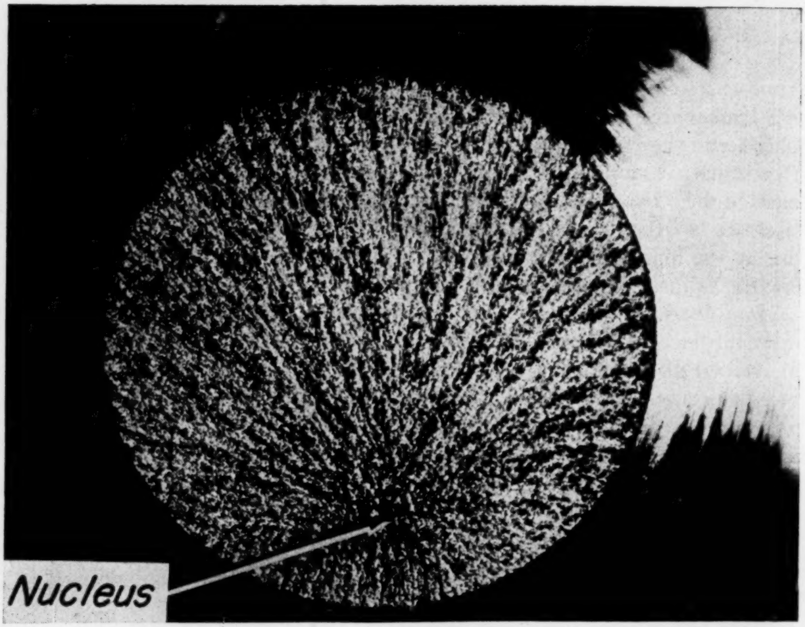
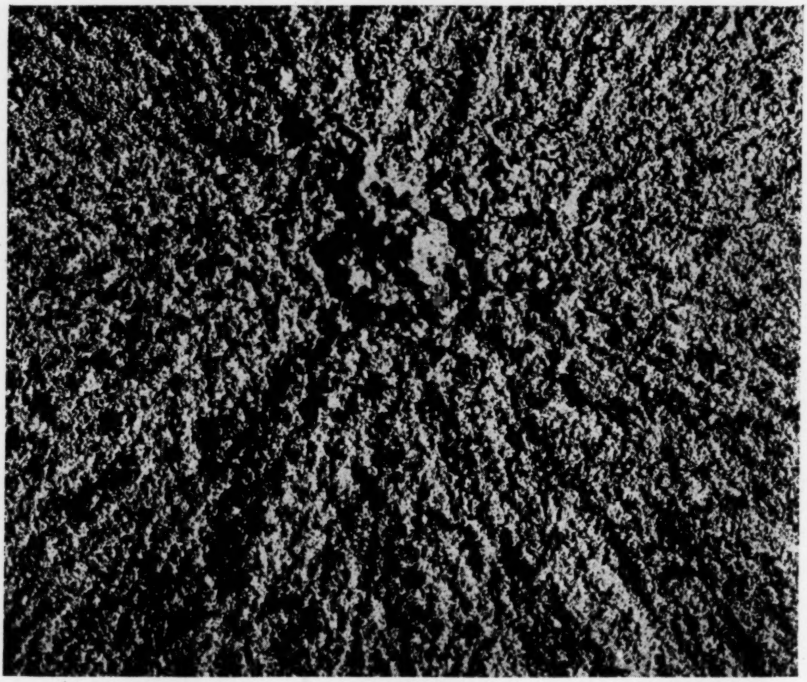
[8] An analysis of fracture photographs by Krafft in support of the postulated surface nucleation of the crack is not presented in his report. Rather demonstration of the proposed surface nucleation of the crack is advanced from fracture load changes with changes in notch root radius. An increase in the notch radius, however, is equivalent to a size reduction for constant notch radius. From the earlier sections of this report this understandably leads to strength modifications due to plasticizing conditions under the notch. The fan pattern development in Krafft's photographs is at variance with the mechanism of fracture propagation attributed to Irwin.

[9] It will be recognized that the surface nucleation of the crack which has been unquestionably observed is proposed to lead to conditions which bring about internal nucleation of the crack which dominates final separation of the section. This question has not been



(*a*) Fracture surface.

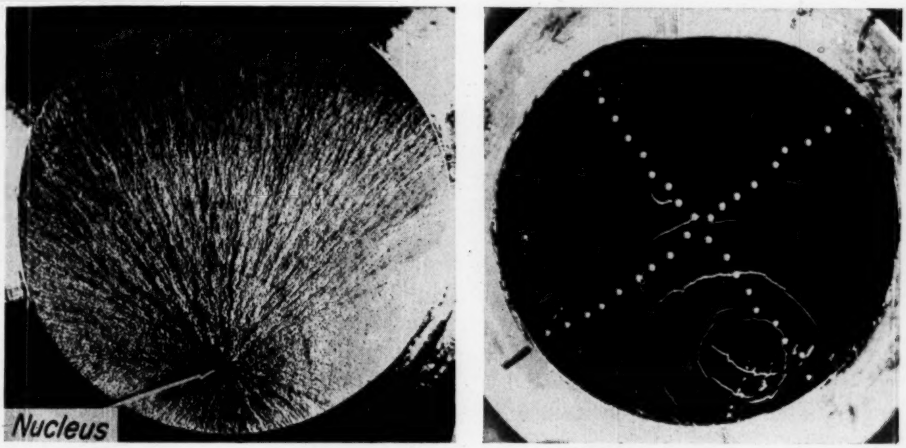


(*b*) Fracture flaw ×10.

FIG. 15.—Fracture Surface of 4340 Steel 1.1-in. Diameter Specimen Illustrating Fracture Development from Internal Flaw; Heat Treatment: 1550 (4) OQ; 400 (1).

The fracture facets presented in Figs. 11 to 14 illustrate certain characteristic and extreme fracture types. In the aligned concentrically loaded notch bar the fracture may be rather generally nucleated and it then develops from a relatively large region. If the ductility of the steel is very low, an internal flaw may be sufficiently severe to initiate the crack that leads to separation of the section. Such a flaw is illustrated in Fig. 15. The general disposition of the fan pattern about the flaw clearly establishes the dominating role played by the flaw in leading to fracture.

One final set of macrographs of the fracture development in a low notch strength specimen is presented in Fig. 16. The fan pattern development in this specimen would require simultaneous radial propagation of the crack along the entire periphery of the notch section. This would seem to be a highly remote possibility, as experimentally it has been shown that the crack tends to run circumferentially; yet in the present investigation two such fractures have been investigated at length.



(*a*) Fracture surface. (*b*) Fracture surface polished.

FIG. 16.—Fracture Surface of 4340 Steel 1.1-in. Diameter Notch Tension Specimen; Heat Treatment: 1550 (4) FC 1350 (1) OQ; 400 (1).

Further evidence that the crack leading to final breaking of the specimen developed from the internal nucleus is offered by examination of the shatter cracks that accompanied crack development. The focal point for the shatter cracks clearly is the point described as the hub of the fan, and it would seem impossible to account for this circular array of shatter cracks in terms of surface nucleation of the fracture.

It is believed, then, that for the concentrically loaded specimens studied in this investigation the fracture process can best be described as taking place in the following way:

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studied directly but is believed to arise as follows: (1) With surface nucleation of the crack a load shift develops. (2) The strains arising from this load cannot be fully offset in the aligning fixture and there is slight bending of the specimen. (3) The bending moment tends to unload the notch adjacent to the internal fracture nucleus—that is, opposite the initial surface fracture nucleus—and the circumferential crack tends not to develop at this position. (4) Due to low ductility in this zone and the conditions of high restraint throughout the section, the probability of internal fracture nucleus development is high and with its development the crack rapidly develops and runs.

1. With increasing load the fracture strain at some chance point on the notch base is exceeded and a surface fracture nucleus develops.

2. If the fracture nucleus is to propagate, this must take place principally along the base of the notch.

3. With surface crack development the load away from the location of maximum penetration of the crack tends to maximize.

4. In this volume of metal, an internal fracture nucleus is formed. It propagates with the development of a characteristic fan and the section is parted.

SUMMARY AND CONCLUSIONS

Circumferentially notched cylindrical tension specimens of different sizes have been concentrically loaded to failure. Materials investigated have been a 4340 steel heat treated in several ways to a range of strength levels, low- and high-strength titanium, and low- and high-strength aluminum. The development of the cracks which lead to failure in the test specimens has been discussed at length. The following conclusions are drawn:

1. For the heat-treated steel, the variations in austenitizing treatment have not led to significant notch properties changes. Tempering time, however, is very important, with reduced times leading to reduced properties.

2. For the three material types studied, notch sensitivity has been shown to depend on the strength level. The size effect phenomenon is not evident for the softer structural conditions examined, while it is present for the harder conditions.

3. In the small specimens fracture is initiated only after significant prior plastic flow. This leads to significant notch geometry changes, which clearly alter stresses resulting from the imposed geometry. The parallel changes in the large specimens are not observed, so the stress conditions in the large specimens must be relatively much more severe than in the small specimens.

4. If in small specimens, notch conditions can be introduced to correspond to those in the large specimens, notch properties corresponding to those in the large specimens should be obtained. Limited examples of such tests are offered.

5. In the circumferentially notched specimens studied, cracking is initiated in the vicinity of the notch root. This crack, while local in origin, tends to propagate around the notch.

6. The circumferential crack leads ultimately to the development of an internal crack which propagates radially to separation of the test section.

Acknowledgment:

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A MODEL FOR THE EFFECT OF THICKNESS ON FRACTURE TOUGHNESS

By Joseph I. Bluhm[1]

Synopsis

A simple model of the fracture phenomenon in thin sheet is suggested based essentially upon the assumption that the shear lip formation is a volume-sensitive mechanism whereas flat fracture is essentially a surface phenomenon. A critical shear lip width is hypothesized; this concept is substantiated and is shown to lead to the variations in shear lip width with thicknesses that were experimentally observed for a beta titanium alloy. Furthermore, the fracture toughness - thickness relationship constructed by the simple linear superposition of the above two fracture mechanisms predicts satisfactorily the observed fracture toughness values for this alloy. A numerical illustration indicates how two tests at different thicknesses might be used to depict the $\mathcal{G}_c$ versus thickness curve at a given temperature over a wide range of thicknesses.

The data of Repko (**1**),[2] although consistent with observed results for thick sheet, are inconclusive with respect to the detailed behavior in the region of 100 per cent shear failures. The work of Irwin (**2**) has aptly demonstrated the influence of thickness over a wide range of thicknesses, and the characteristic peaked fracture toughness, $\mathcal{G}_c$ versus reciprocal thickness $1/t$, curve observed is reproduced in Repko's Fig. 9(*a*). The limited decrease in $\mathcal{G}_c$ with decrease in thickness noted in Irwin's work is not evident in Repko's work (**1**), possibly because Repko did not investigate sufficiently thin sheet. Others (**3–7**) also have observed this characteristic (peaked) shape of the $\mathcal{G}_c$ versus thickness t (or reciprocal thickness) curve, and it appears certain that this shape is a consequence of changes of fracture mode with thickness.

Irwin (**8**) has attempted to rationalize qualitatively on the mechanism associated with the transition from shear lip to flat fracture and has suggested a criterion based upon the relative size of the plastic zone at the crack tip and the thickness of the plate.

In spite of the considerable activity in the fracture mechanics area, no attempt appears to have been made to establish rationally a basis for anticipating this experimentally observed characteristic shape of the $\mathcal{G}_c$ versus t relationship. Nevertheless such a basis can be quite simply formulated with a minimum of conceptual requirements.

Assumptions

It is assumed for purposes of the present analysis that:

1. The total fracture toughness (integrated over the fracture surface)

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[1] Chief, Materials Engineering Laboratory, Watertown Arsenal Laboratories, Watertown, Mass.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

derives from a dual fracture mode mechanism, that is, either combinations of flat fibrous break and shear lip or flat cleavage break and shear lip.[3]

2. The shear lip occupies the full sheet thickness (for a given temperature) until a critical thickness t_0 is reached; beyond this critical sheet thickness the total lip width no longer increases; Fig. 1 indicates schematically the fracture model assumed for the present analysis.

3. The shear lip, once fully developed in accordance with assumption 2 above remains constant in width along the length of the fracture path. For lip length to lip width ratios ≫ 1 extensive data (reference (**3**, Fig. 11); Wessel (**4**, Fig. 7); Srawley and Beachem (**5**, Fig. 2); and Irwin and Kies (**9**, Fig. 4)) appear to substantiate this assumption. Deviation from this assumption may, of course, be expected at the point of onset or arrest of the crack, that is, at the points where end effects are significant. Charpy bars, for example, tend to exaggerate the effects of end condition. Repko et al (**1**), on the other hand, do indicate one condition of one material examined in which even for a long lip the shear lip width varies from 30 to 70 per cent along the fracture path. However, this is characteristic of an extremely rough coarse grained fracture for the material machined from a single heat and is not typical of most alloys. The contention of constancy of lip width appears nevertheless to be further supported by the results of limited tension tests of as-received AISI 4340 specimens. A specimen 3.57 in. in diameter was tested in tension to fracture. Figure 2(*a*) shows the fracture surface and depicts an eccentrically located relatively large area of slow crack growth. The average shear lip width is of the order of 0.030 ± 0.005 in. The value of shear lip width was determined by averaging the widths taken at 15 deg intervals about the circumference. The individual readings were obtained using an optical comparator. Subsequent to this large-specimen test, a

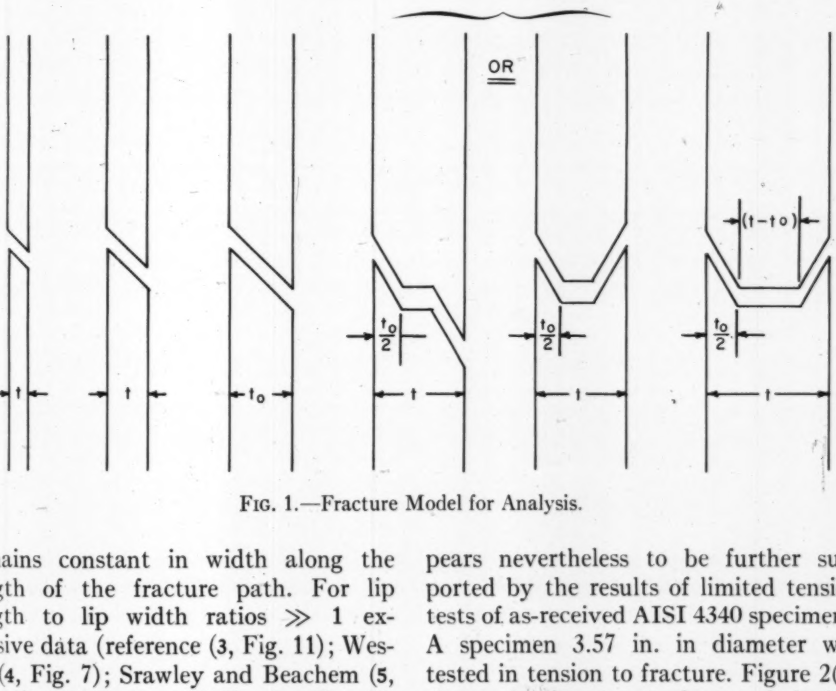

FIG. 1.—Fracture Model for Analysis.

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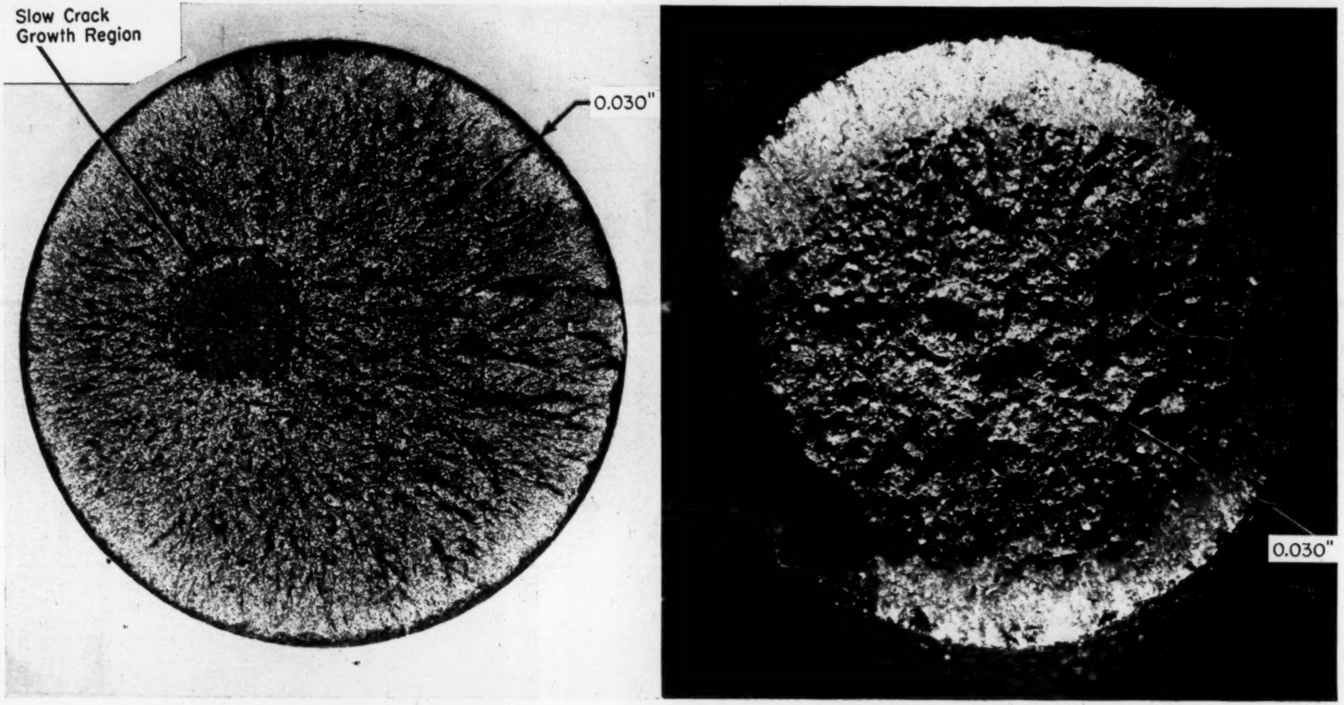
[3] Some modification is to be made to extend the present consideration to those materials where a combination of three modes of fracture, that is, flat fibrous, flat cleavage, and shear lip, may develop in significant proportions simultaneously.


Slow Crack
Growth Region
0.030"
0.030"

(*a*) 3.57 in. diameter specimen (×1). (*b*) 0.357 in. diameter specimen (×10).

FIG. 2.—Fracture Surfaces of AISI 4340 (As-received) Illustrating the Concept of a Critical Shear Lip Width.

standard (0.357-in. diameter) specimen was machined from a longitudinal slice through a threaded region of one half of the larger specimen, and tested. Figure 2(*b*) shows this fracture. This specimen size was such as to limit crack growth to only slow propagation. Nevertheless the lip size in this specimen, measured in the same means as the larger specimen, was 0.035 ± 0.005 in.

It is of further interest to note that this critical lip width appears to be relatively insensitive to the character of the flat break from which it proceeds. For example, in the large specimen the lip formation follows a "rapid" flat fracture whereas in the small specimens (whose diameter is less than the critical crack size indicated by the "slow" growth region of the larger specimen; see Fig. 2(*a*)) the lip forms as the final stages of a slow flat fracture. Nevertheless the lip width in these two cases is not significantly different. More general substantiation of this superficial observation is, of course, desirable.

The validity of this assumption is further demonstrated by comparing the curve of Fig. 8(*b*) of Repko, Jones, and Brown (1) with the corresponding curve calculated[4] upon the basis of this assumption. Figure 3 shows the original data of Repko et al (1) (as well as his curve through these points) and the calculated curve. The agreement is remarkable and supports the contention of a constant shear lip width after full development.

4. The mechanism of flat fracture is essentially a surface phenomenon whereas the mechanism of lip formation is volumetric in nature; the energy of flat fracture is assumed to be proportional to the width of the flat area, that is, to $(t - t_0)$ of Fig. 1; the energy of shear lip fracture is assumed to be proportional to the lip width $t/2$ squared until the lip is fully developed to width $t_0/2$ and is then a constant.

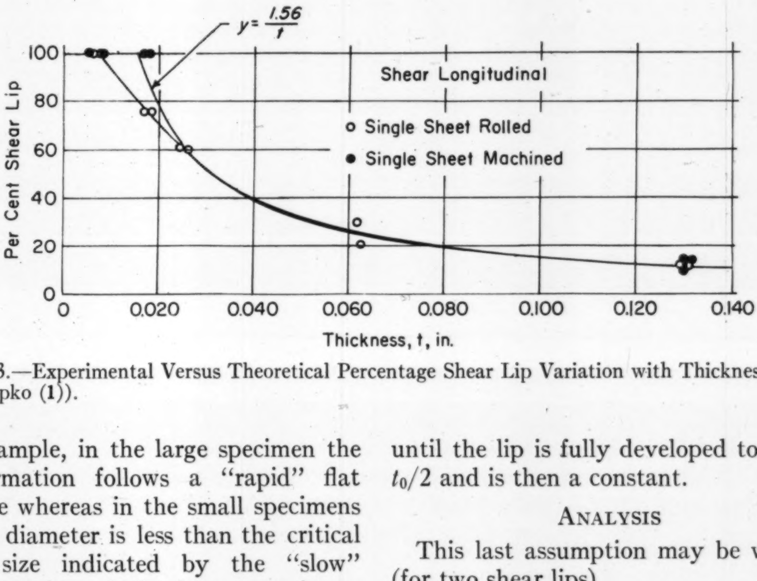


FIG. 3.—Experimental Versus Theoretical Percentage Shear Lip Variation with Thickness (Data after Repko (1)).

ANALYSIS

This last assumption may be written (for two shear lips)

$$\left.\begin{array}{ll} E_{sl} = \frac{1}{2}k_{sl}t^2l & t \leq t_0 \\ \text{and} & \\ \left.\begin{array}{l} E_{sl} = \frac{1}{2}k_{sl}t_0^2l \\ E_{ff} = k_{ff}(t - t_0)l \end{array}\right\} & t \geq t_0 \end{array}\right\} \quad \ldots\ldots(1)$$

---

[4] For example, from Fig. 8(*b*) of Repko, Jones, and Brown (**1**) we note that for $t = 0.130$ the percentage shear lip is 12 per cent; the fully developed or critical shear lip t_0 is then given by $t_0 = 0.12 \times 0.130 = 0.0156$ in. The present assumption is that as long as the plate is as thick as or thicker than t_0 (0.0156 in.), the lip width will remain just 0.0156 in. Then in terms of a plate of arbitrary thickness $t \geq t_0$, the percentage shear lip (per cent *SL*) is then per cent $SL = 100\, t_0/t = 1.56/t$.

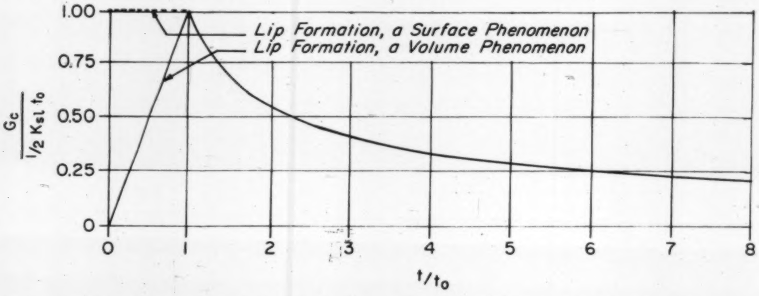


FIG. 4.—Predicted Variation of $\mathcal{G}_c$ with Sheet Thickness Normalized with Respect to $\frac{1}{2}k_{sl}t_0$ $k_{ff} = \frac{1}{20} k_{sl}t_0$) (from Eq 5).

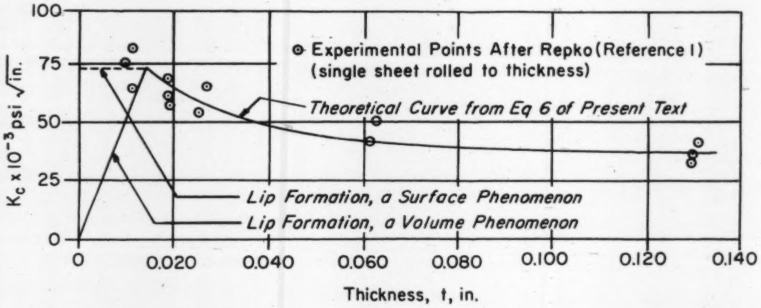


FIG. 5.—Comparison of Predicted and Experimental Fracture Toughness Versus Thickness.

where the E's are the total energies and the subscripts sl and ff refer to shear lip and flat fracture respectively. The k's are material constants associated with the mode of fracture; t and t_0 are the thickness and critical thickness for 100 per cent shear lip respectively, and l is a characteristic length determining the total energy.

With these definitions one can write for the total energy E_T the following:

$$E_T = E_{sl} + E_{ff} \qquad (2)$$

Now the total energy per unit nominal fracture area which we may recognize as being identically equal to $\mathcal{G}_c$ is then

$$\mathcal{G}_c = E_T/lt \qquad (3)$$

where lt is the fracture area. Then by substituting Eqs 3 and 1 into Eq 2 we find

$$\left.\begin{aligned} &\mathcal{G}_c = \tfrac{1}{2}k_{sl}t_0(t/t_0) && \text{for } t/t_0 \leq 1 \\ &\text{and} \\ &\mathcal{G}_c = \tfrac{1}{2}k_{sl}t_0(t_0/t) + k_{ff}(1 - t_0/t) \\ & && \text{for } t/t_0 \geq 1 \end{aligned}\right\} \qquad (4)$$

Normalizing with respect to $\frac{1}{2}$ $k_{sl}t_0$, these become

$$\left.\begin{aligned} &\mathcal{G}_c/\tfrac{1}{2}k_{sl}t_0 = (t/t_0) && \text{for } t/t_0 \leq 1 \\ &\text{and} \\ &\mathcal{G}_c/\tfrac{1}{2}k_{sl}t_0 = \frac{1}{(t/t_0)} + \frac{k_{ff}}{\frac{1}{2}k_{sl}t_0}\left(1 - \frac{1}{t/t_0}\right) \\ & && \text{for } t/t_0 \geq 1 \end{aligned}\right\} \qquad (5)$$

If we now further assume (strictly for illustrative purposes) that $k_{ff} = \frac{1}{20}$ $k_{sl}\,t_0$, then these relations may be readily computed. Figure 4 shows the results of such a calculation. Except for the sharp maximum, this is a remarkably good first approximation of the observed characteristic behavior. End effects, referred to under assumption 3, will tend to smooth the curve.

In terms of the stress parameter K_c, defined (3) by the relation

$$K_c^2 = E\mathcal{G}$$

where E is the Young's modulus, Eq 4 may be rewritten

$$\left.\begin{aligned} K_c &= \sqrt{E\tfrac{1}{2}k_{sl}t} \qquad \text{for } t/t_0 \le 1 \\ \text{and} & \\ K_c &= \sqrt{E[\tfrac{1}{2}k_{sl}t_0(t_0/t) + k_{ff}(1 - t_0/t)]} \\ & \qquad \text{for } t/t_0 \ge 1 \end{aligned}\right\} \ldots(6)$$

It is of interest to observe that if the shear lip formation mechanism is not volumetric in nature but is rather a surface phenomenon, then in the 100 per cent shear region of thicknesses the $\mathcal{G}_c$ value will be a constant as shown by the dashed lines of Figs. 4 and 5. Obviously Repko's data plotted in Fig. 5 do not resolve this possible difference. However, data by Arnold (7) and Irwin (2) definitely tend to substantiate the volumetric concept.

It is further interesting to note that for $t \ge t_0$, Irwin (2) has suggested a relationship which is equivalent to

$$\frac{\mathcal{G}_c - k_{ff}}{k_{ff}} \propto \left(\frac{1}{t}\right)^2 \quad \text{where } k_{ff} = \mathcal{G}_{cI}$$

whereas Eq 4 leads to the conclusion that

$$\frac{\mathcal{G}_c - k_{ff}}{k_{ff}} \propto \left(\frac{1}{t}\right).$$

Additional experiments may resolve this difference.

EVALUATION OF THE MATERIAL CONSTANTS k_{sl}, k_{ff}, AND t_0

Practical application of the foregoing results, Eqs 4 or 5, can be effected by conducting and evaluating the results of only two sets of notch tests from which the three unknown quantities k_{sl}, k_{ff}, and t_0 may be determined.

Consider the test results for sheet thickness t_1 and t_2 where $t_2 > t_1$, and $t_2 > t_0$, that is, where the thicker specimen is at least thick enough to produce a partially flat fracture. Let $\mathcal{G}_{c_1}$ and $\mathcal{G}_{c_2}$ be the corresponding fracture toughness determination and let per cent SL_2 be the per cent shear lip observed in the thicker specimens. The critical shear lip width t_0 is then determined according to the prior footnote, thus in effect reducing Eq 4 to a set of two equations in the two unknowns k_{sl} and k_{ff}.

Then the last of Eq 4 may be written as follows:

$$\left.\begin{aligned} \mathcal{G}_{c_1} &= \tfrac{1}{2}k_{sl}\frac{t_0^2}{t_1} + k_{ff}\left(1 - \frac{t_0}{t_1}\right) \\ \text{and} & \\ \mathcal{G}_{c_2} &= \tfrac{1}{2}k_{sl}\frac{t_0^2}{t_2} + k_{ff}\left(1 - \frac{t_0}{t_2}\right) \end{aligned}\right\} \ldots(7)$$

whence by simultaneous solution we obtain

$$k_{sl} = \frac{\begin{vmatrix} \mathcal{G}_{c_1} & (1 - t_0/t_1) \\ \mathcal{G}_{c_2} & (1 - t_0/t_2) \end{vmatrix}}{\Delta};$$

$$k_{ff} = \frac{\begin{vmatrix} \tfrac{1}{2}t_0^2/t_1 & \mathcal{G}_{c_1} \\ \tfrac{1}{2}t_0^2/t_2 & \mathcal{G}_{c_2} \end{vmatrix}}{\Delta};$$

$$\Delta = \begin{vmatrix} \tfrac{1}{2}t_0^2/t_1 & (1 - t_0/t_1) \\ \tfrac{1}{2}t_0^2/t_2 & (1 - t_0/t_2) \end{vmatrix} \ldots(8)$$

For any thickness t then Eqs 4 apply.

Note that for thick sheet, that is, $t_0/t \ll 1$, $\mathcal{G}_c$ approaches $k_{ff} = \mathcal{G}_{cI}$ and that the maximum value of $\mathcal{G}_c$ occurs at $t/t_0 = 1$ and is given by

$$\mathcal{G}_{c\max} = \tfrac{1}{2}k_{sl}t_0$$

NUMERICAL ILLUSTRATION (Based upon Data by Repko et al (1) for a Beta Titanium Alloy)

From the data of Repko (1) (Figs. 8(*a*) and (*b*) of their paper), we note the results for $t_1 = 0.045$ and $t_2 = 0.130$, as follows, along with the derived values of $\mathcal{G}_c$ and t_0 :

t, in.	k_c, psi $\sqrt{\text{in.}}$	Per Cent SL	$\mathcal{G}_c$,[a] in-lb per sq in.	t_0,[b] in.
$t_1 = 0.045$	50,000	..	(156.5)	...
$t_2 = 0.130$	38,000	12	(92)	0.0156

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[a] Plane stress assumed for the conversion.

[b] t_0 = per cent $SL_2 \times t_2/100 = 0.12 \times 0.130 = 0.0156$.

Then from Eq 8

$$\begin{aligned} \tfrac{1}{2}k_{sl} &= 21{,}800.0 \\ \tfrac{1}{2}k_{sl}t_0 &= 339.0\ (\mathcal{G}_{c\,\max}) \\ k_{ff} &= 59.5\ (\mathcal{G}_{cI}) \end{aligned}$$

whence finally Eq 4 may be written

$$\mathcal{G}_c = 21{,}800t \qquad \text{for } t \leq 0.0156$$

$$\mathcal{G}_c = 339.0\left(\frac{t_0}{t}\right) + 59.5\left(1 - \frac{t_0}{t}\right) \qquad \text{for } t \geq 0.0156$$

or

$$K_c = 5.9 \times 10^3\sqrt{t} \qquad \text{for } t \leq 0.0156$$

and

$$K_c = 4.0 \times 10^3\sqrt{339t_0/t + 59.5(1 - t_0/t)} \qquad \text{for } t \geq 0.0156$$

Figure 5 shows a plot of these equations superimposed on the experimental data. Again the agreement appears entirely adequate for $t \geq t_0$ but is questionable in the region of $t \leq t_0$.

CONCLUSIONS

On the basis of the limited tests described, it is concluded that:

1. The per cent shear lip variation with sheet thickness is reasonably well predicted on the basis of the result of a single thick specimen and the critical lip width concept.
2. The $\mathcal{G}_c$ or K_c thickness curve reported in the literature is reasonably well predicted for one heat of beta titanium, at least in the region of $t \geq t_0$, on the basis of the simple dual fracture mode model and the principle of superposition of fracture energies.
3. In the region of $t \leq t_0$ the volumetric sensitive shear lip formation mechanism leads to the $\mathcal{G}_c$ or K_c versus thickness trends observed by several investigators; this mechanism is not conclusively demonstrated by the data of Repko.
4. Two $\mathcal{G}_c$ tests (at a given temperature) are sufficient to calculate the material constants necessary for determining the $\mathcal{G}_c$ or K_c versus thickness curve.

Acknowledgment:

The author is indebted to Mr. F. Larson of the Watertown Arsenal Laboratories for his detailed observations of the mechanism of lip formation which prompted the author to modify Fig. 1 and for his courtesy in supplying the 10× steel tension specimen referred to in the text.

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A MODEL FOR THE EFFECT OF THICKNESS ON FRACTURE TOUGHNESS APPLIED TO LAMINATED STRUCTURES

BY JOSEPH I. BLUHM[1]

SYNOPSIS

A model previously suggested for predicting the fracture behavior of thin sheet is extended to cover laminated composites. Thus extended, this model leads to a prediction of the behavior of laminated Charpy bars which is at least qualitatively substantiated by the observed behavior.

Arnold (**1**)[2] has proposed a laminated Charpy specimen for evaluating impact strength of sheet metal, and has experimentally determined the effect of sheet thickness on that impact strength. In the present paper it is suggested that the behavior of such laminates may be predicted on the basis of a model proposed by Bluhm (**2**).

TOTAL ENERGY ABSORPTION—SINGLE LAMINA

Bluhm's (**2**) model leads to the formulation:

$$\left.\begin{array}{ll} E_T = \tfrac{1}{2}k_{sl}t^2l & \text{for } t \leq t_0 \\ \text{and} & \\ E_T = \tfrac{1}{2}k_{sl}t_0^2l + k_{ff}(t - t_0)l & \text{for } t \geq t_0 \end{array}\right\} \ldots(1)$$

where k_{sl}, k_{ff} and t are assumed to be material properties,[3] t_0 specifically is the critical shear lip width as defined in reference (**2**), E_T is the total energy absorption, l is the length of the shear lip (in the present discussion as relating to thin Charpy bars, l is taken as the depth of the Charpy bar below the notch), and t is the thickness of the sheet.

A plot of these relations is shown schematically in Fig. 1 for two alternative cases. It is to be emphasized that the straight-line segment of this relationship (stage *II* of Fig. 1) does not in general intersect the coordinate origin. Furthermore, for thin sheet, that is, where $t < t_0$, the relationship is nonlinear (stage *I* of Fig. 1). Data by Arnold (**4**) for AISI 4340 steel at 165,000 and 210,000 psi tensile strength tend to support this latter observation.

EVALUATION OF $\mathcal{G}_{cI}$ (PLANE STRAIN FRACTURE TOUGHNESS)

If the second part of Eq 1 is divided by l and differentiated with respect to thickness, we obtain:

$$\frac{dE_T}{l\,dt} = k_{ff} \ldots\ldots\ldots\ldots\ldots(2)$$

But k_{ff} is in effect identically $\mathcal{G}_{cI}$. Therefore, we note that the slope of the total energy E_T thickness t curve (in the region $t \geq t_0$) divided by the shear lip length l is a direct measure of $\mathcal{G}_{cI}$. Though some uncertainty may arise from the selection of a value for l, the shear lip length, if l is such that $l \gg t_0$, the use of the total specimen depth or total shear lip length is expected to provide a reasonable approximation of $\mathcal{G}_{cI}$.

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[1] Chief, Materials Engineering Laboratory, Watertown Arsenal Laboratories, Watertown, Mass.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

[3] Curll (**3**) has suggested a somewhat similar relationship governing the size effect in Charpy bars.

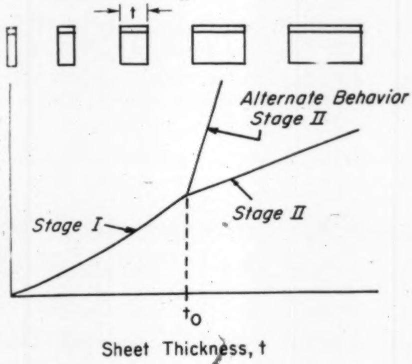

FIG. 1.—Schematic Representation of Predicted Energy Versus Thickness Relation (per Eq 1).

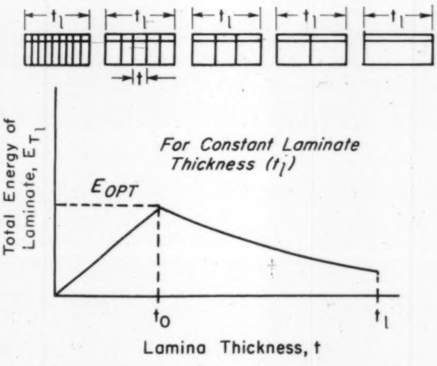

FIG. 2.—Schematic Representation of Predicted Laminate Energy Versus Lamina Thickness (per Eq **4**).

TOTAL ENERGY ABSORPTION—MULTIPLE LAMINAE

Constant Laminate Thickness:

Consider now a laminate. If the interaction between laminae of a laminated structure is neglected, that is, if each lamina acts independently, then Eq 1 may be expressed for a laminated specimen as follows:

$$\left.\begin{aligned} E_{Tl} &= \frac{n}{2} k_{sl} t^2 l \qquad \text{for } t \le t_0 \\ &\text{and} \\ E_{Tl} &= \frac{n}{2} k_{sl} t_0^2 l + n k_{ff}(t - t_0) l \qquad \text{for } t \ge t_0 \end{aligned}\right\} \quad \ldots (3)$$

where now E_{Tl} is the total energy absorbed in a laminated specimen having n laminae, each of thickness t.

For a fixed laminate thickness $t_l = nt$, Eq 3 may be expressed in the form

$$\left.\begin{aligned} E_{Tl} &= \frac{t_l}{2} k_{sl} t l \qquad \text{for } t \le t_0 \\ &\text{and} \\ E_{Tl} &= \frac{t_l k_{sl} t_0^2 l}{2t} + t_l k_{ff} \left(1 - \frac{t_0}{t}\right) l \\ &\qquad \text{for } t \ge t_0 \end{aligned}\right\} \quad \ldots (4)$$

Figure 2 shows these relationships schematically. It will be noted that an optimum lamina thickness exists and is just equal to the critical thickness t_0. The maximum total energy absorption corresponding to a laminate made up of n laminae each of critical thickness t_0 is designated in Fig. 2 as the "optimized" energy absorption E_{opt}. This analytical prediction of an optimum lamina thickness to maximize the laminate energy absorption is in accordance with certain observations of Arnold as indicated on p. 3 of reference (**4**) and in Fig. 2 of reference (**5**).

On the other hand, the foregoing theory and observations by Arnold appear to conflict with respect to the single lamina behavior. The theory based on the suggested model predicts the two-stage behavior illustrated in Fig. 1, that is, a nonlinear stage I and then a linear stage II with a monotonically increasing energy absorption with increased sheet thickness. Arnold's results, Fig. 2A of reference (**5**), imply an optimum lamina thickness to achieve maximum energy absorption in individual lamina. The basis for these discrepancies is not completely understood, though it is possible that the energy absorption shown by Arnold (Fig. 2A, reference (**5**)) for the thin specimen may be exaggerated due to the interference effects of individual lamina on one another as they tend to expand laterally. This hypothesis cannot be verified from the data given by Arnold (**5**,**6**) inasmuch as his single sheet specimens are not of the same material as his laminate constituents. Supplementary tests are therefore desirable. Other data by Arnold on 2024 T-4 aluminum alloy do in fact corroborate the Stage *II* behavior anticipated in Fig. 1 for individual lamina (see, for example, Fig. 7 of reference (**6**)).

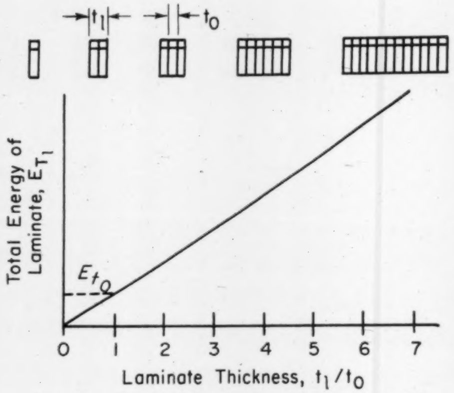

FIG. 3.—Schematic Representation of Predicted Energy Versus Thickness Relation of "Optimized" Laminate (per Eq **5**).

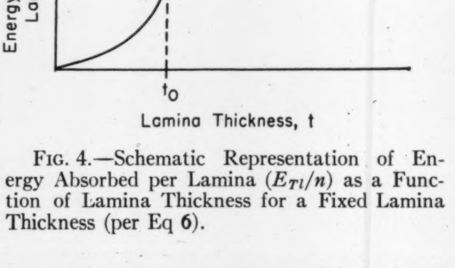

FIG. 4.—Schematic Representation of Energy Absorbed per Lamina (E_{Tl}/n) as a Function of Lamina Thickness for a Fixed Lamina Thickness (per Eq **6**).

Variable Laminate Thickness:

If the laminate thickness[4] is considered a variable, then optimization with respect to total energy absorption can be effected by building up the laminate with n laminae, each of critical thickness t_0. From Eq 3, for example, we obtain (if each lamina is of critical thickness t_0):

$$E_{\text{opt}} = n \frac{k_{sl}}{2} t_0^2 l \qquad (5)$$

Figure 3 shows schematically this linear relation between the total energy absorption of an "optimized" laminate versus the laminate thickness expressed as $n = t_l/t_0$. Total thickness is governed, of course, by other strength requirements.

ENERGY ABSORPTION PER LAMINA

The energy per lamina E_{Tl}/n is readily shown to be given by

$$\left.\begin{array}{ll} E_{Tl}/n = \frac{1}{2}k_{sl}t^2 l & \text{for } t \le t_0 \\ \text{and} & \\ E_{Tl}/n = \frac{1}{2}k_{sl}t_0^2 l + k_{ff}(t - t_0)l & \text{for } t \ge t_0 \end{array}\right\} \qquad (6)$$

These relations are plotted schematically in Fig. 4. The ordinate E_{Tl}/n represents the energy absorbed in a single lamina of a laminate (the shaded region of the specimens shown). Note that these relations, as should be expected, are identically the same as for the single lamina results of Eq 1, or Fig. 1.

CONCLUSIONS

It is evident from these studies that:

1. The model suggested is capable of describing, at least qualitatively, the behavior of individual lamina as well as

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[4] Laminate thickness is assumed for illustration to be an integral multiple of t_0.

laminates insofar as the effect of thickness on energy absorption is concerned.

2. The experimentally observed existence of a lamina thickness optimized for maximizing the energy absorption in a laminate is theoretically supported; this optimum thickness is the critical thickness t_0 (see, for example, Fig. 2).

3. Additional tests need to be conducted wherein both laminates and laminae are tested in order to resolve the discrepancies still evident.

DEFINITION OF SYMBOLS

t_l = laminate thickness,

t = lamina thickness,

t_0 = critical lamina thickness for 100 per cent shear,

n = number of laminae in laminate,

E_T = total energy absorption of a lamina,

E_{Tl} = total energy absorption of a laminate,

k_{sl}, k_{ff} = material constants associated with shear lip and flat fracture respectively,

E_{opt} = total energy absorption of laminate made of laminae, each of thickness t_0 (optimized total energy absorption), and

l = the length of the shear lip.

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POTENTIAL HEAT*

A METHOD FOR MEASURING THE HEAT RELEASE OF MATERIALS IN BUILDING FIRES

BY J. J. LOFTUS,[1] D. GROSS[1], AND A. F. ROBERTSON[1]

SYNOPSIS

Modifications have been made of a method, currently a standard of the French government, for assessment of the combustible characteristics of building materials. This method makes use of standard calorimetric techniques in which the burning of small quantities of combustible in an otherwise inert material is assured by use of a combustion promoter which is added prior to test. By performing calorimetric measurements both before and after exposure to a "standardized fire" (2 hr in air at 750 C) the difference may be considered as the potential heat. The test method has been used for measurement of the potential heat of a variety of building materials, including metals and materials of low combustibility, with results in general agreement with fire experience. A discussion is included on the precision of the test method.

The extent to which building materials burn and release heat during fires involving buildings is considered of importance by code officials. Because of this, building codes generally contain requirements with respect to the "noncombustible" properties of materials to be used in different portions or types of buildings. However, the test methods (**1, 2, 3, 4, 5**)[2] most frequently proposed or used for measuring this property have been qualitative in nature. The results obtained have often been a function of the flammable, smouldering, and heat release characteristics of the material tested with emphasis varying, depending on the characteristics of the material and the test method used. As a result, there has been considerable uncertainty as to exactly what material property was being measured. Because of this situation, it has been difficult to select a method of test which could be considered satisfactory for standardization on a national or international level.

To reduce the complexities of the problem, it is convenient and indeed necessary to separate the surface flammability from the "combustible" properties in the proper evaluation of building materials. Whereas surface flammability is concerned with the rate with which flames travel along the surfaces of materials, it is the extent to which the bulk of a wall, ceiling or floor element may contribute heat to support active combustion which represents the degree of its combustibility or, perhaps more appropriately, its "potential heat."

Of the actual combustibility test

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[2] The boldface numbers in parentheses refer to the list of references attached to this paper.

methods in use, considerable attention has been given to the one in use in the United Kingdom (4). By this method, a sample is gradually heated during the course of 1½ hr in a vertical electrically heated furnace up to a temperature of 750 C, and observations made of the occurrence of flame, self-induced glow, or of gases that can be ignited by a pilot flame. In a common modification of the method, the sample is placed into a preheated furnace maintained at 750 C, and a record obtained of the temperatures on the surface and in the interior of the specimen. In the usual case, a limitation on the maximum temperature rise of the sample above the furnace temperature is used as a criterion of incombustibility. A modification of this method (6) involves the measurement of the area under the temperature-time curve for the center of the sample. However, Van Elteren (7) has indicated that neither the temperature rise nor the temperature-time product is a direct criterion of the heat of combustion. This results from the transient nature of the test and the strong dependence of the temperature-time history on the thermal properties of combustible and filler materials. In effect, this heated tube test method does not provide a good measure of the quantity of heat released by the material.

To evaluate properly the total heat generated by the combustible fraction of a material, calorimetric test methods of various types have been considered. Factory Mutual Laboratories has recently described a calorimetric furnace by means of which measurements are made of heat release rates for 4-ft square test specimens of constructions (8). Bomb combustion calorimetric measurements have also been considered, but in the past these have been rejected because the combustion characteristics in an oxygen pressurized bomb are much different from those effective during building fires. For example, the bomb calorimeter results in complete oxidation of many metals which are relatively unaffected by building fires. However, the French government (9) has recently standardized a test method making use of a differential bomb calorimetric method.

The present paper describes the further development of a similar test procedure and presents the results of measurements of the potential heat of a wide variety of building materials. The potential heat of a material may be defined as the difference between the heat of combustion of a representative sample of material and the heat of combustion of the residue remaining after exposure to a simulated standard fire, using combustion calorimetric techniques.

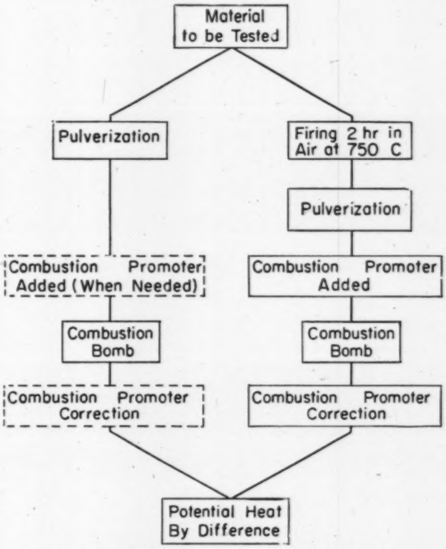
Material to be Tested
Pulverization
Firing 2 hr in Air at 750 C
Pulverization
Combustion Promoter Added (When Needed)
Combustion Promoter Added
Combustion Bomb
Combustion Bomb
Combustion Promoter Correction
Combustion Promoter Correction
Potential Heat By Difference

FIG. 1.—Schematic Flow Diagram for Potential Heat Measurements.

TEST METHOD

A schematic flow diagram of the test method, Fig. 1, illustrates the steps involved. Two samples are removed from the material to be tested. One of these is pulverized, pelleted, and then burned in a high-pressure oxygen atmosphere as described in reference (**10**), yielding a measure of the gross heat of combustion of the material.[3] A second specimen of approximately $\frac{1}{2}$ by $\frac{3}{4}$ by 3-in. size is "fired" in a muffle furnace. By "firing" is meant heating in air for 2 hr at a temperature of 750 C. The weight loss of this specimen is noted and the residue remaining is ground or pulverized. A portion of this resulting powder corresponding to a known weight of the original material is mixed with a combustion promoter, pelleted, and burned in the combustion calorimeter as previously described. After correcting for the heat produced by the combustion promoter, the difference in heating values of the two specimens is reported as the potential heat. It is evident from this diagram that the potential heat measurement is not a direct one but is obtained by difference measurements. It is indicative of the quantity of heat released during the firing process in the muffle furnace; this exposure, in effect, represents a simulated standard fire.

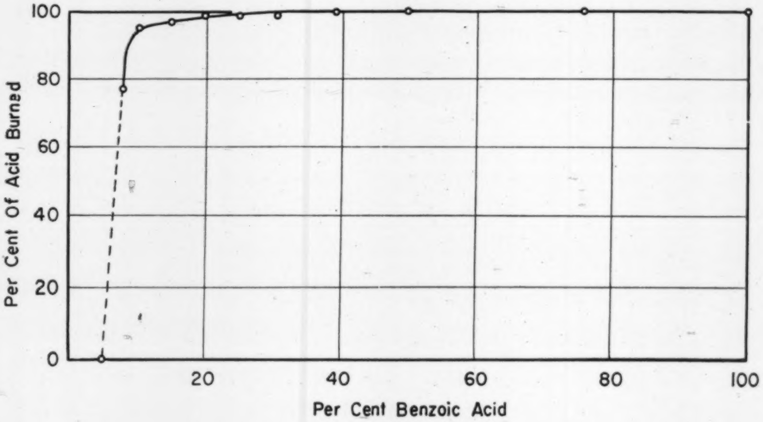

FIG. 2.—Per Cent Acid Burned in Mixtures of Benzoic Acid and Aluminum Oxide.

PROCEDURAL DETAILS

The detailed techniques which have been developed for standardization of the test method are described in the Appendix. A brief discussion will be presented in this section of the reasons for selection of these details.

Firing Conditions:

The conditions under which the specimen is heated in the muffle furnace have an influence on the measurements made. The temperature of 750 C was selected because of its long use in this and other countries for exposure of the specimen in combustibility test methods. The temperature is lower than the maximum which would be expected in fires of even brief duration. It is, however, high enough to permit the ignition of magnesium. Aluminum, on the other hand, will melt and not become inflamed. These results are believed typical of actual fire experience. The selection of specimen size, air feed rate, and duration of high-temperature exposure also insured complete combustion of the fuel fraction in cinder concrete specimens and the oxidation of carbon resulting from pyrolysis of wooden specimens.

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[3] In the case of materials of low combustible content, a combustion promoter is added prior to pelleting the specimen for test in the combustion calorimeter.

Combustion Promoter:

The use of an additive to the specimen prior to testing in the bomb is intended to insure that burning of the combustible content is complete even when present in small quantities. The additive used was benzoic acid of known heating value (11). Determination of the minimum quantity of acid required was made by measurement of the percentage of acid consumed when mixtures of benzoic acid and aluminum oxide were burned in the combustion calorimeter. The results are shown in Fig. 2. It is evident that when the quantity of acid in the sample is less than about 30 per cent, combustion may be incomplete and reliable results should not be expected. Because of this, materials suspected or known to have low combustible content are tested after addition of about 50 per cent benzoic acid. Acid additions are not required with woods, some plastics, and other materials involving high combustible content. The addition of a combustion promoter is always necessary for tests involving the residue fraction.

Specimen Preparation:

The material to be tested was pulverized to insure complete combustion and also to permit incorporation of the combustion promoter prior to pelleting, when necessary. This was accomplished by means of a hand mill for all but the hardest materials. A mortar and pestle as well as files or rasps have been found useful for some otherwise difficult materials.

PRECISION AND ACCURACY OF METHOD

Standard combustion calorimetric techniques would be expected to yield highly precise measurements of the heating values of fuels. Perhaps precisions on the order of 30 Btu per lb would be normal for good quality work with standard laboratory equipment and specimen weights of approximately 1 g. Such precisions should not be expected with the test procedure described here. Not only are differences of two measurements reported, but the method involves mixing a combustion promoter with the specimen as well as the additional complexity of accounting for weight changes resulting from the specimen exposure within the muffle furnace. As a result, errors of somewhat greater magnitude are to be expected.

One way of exploring the precision of the method is to compare the difference between duplicate measurements of the individual calorimetric determinations with the mean heat of combustion values on the unfired specimens. It was found that the differences between duplicates were essentially independent of the heat of combustion and had an average value of 100 Btu per lb. Similar treatment of the fired specimens yielded an average difference of 34 Btu per lb. These averages correspond to standard deviations of 89 and 30 Btu per lb for individual unfired and fired specimens, respectively, or about 63 and 21 Btu per lb for the average of two determinations. The precision of the potential heat measurement would then be the square root of the sum of the squares of these two or about 94 Btu per lb for one and 66 Btu per lb for the average of two determinations.

As a means for demonstrating the sensitivity and usefulness of this method of measuring the heat release potential of building materials, a series of tests was performed with the use of plaster of Paris specimens to which various additions of charcoal had been made. The mixtures of plaster and charcoal were made in the dry state. These were then hydrated and the hardened mixtures used for potential heat determinations. From analysis of the plaster the hemihydrate ($CaSO_4 \cdot \frac{1}{2}\ H_2O$) content was determined as equal to 94 per cent. Computed values of the potential heat were prepared and are presented in Table I which provides an indication of the magnitude of errors in both precision and accuracy. Here, the apparent error in measurement is reported as the deviation of the experimentally determined value from the computed value of potential heat. These errors appear to be of a random nature (see Fig. 3). It is therefore evident that under the conditions of the test complete combustion was achieved in the calorimeter even for mixtures of very low potential heat. It can be shown from these data together with other data on different types of materials containing carbon in known or estimable percentages that the heat of combustion of the carbon fraction in the material is independent of the amount or nature of the combustible or of the inert filler.

TABLE I.—COMPARISON OF COMPUTED[a] AND MEASURED[b] POTENTIAL HEAT OF MIXTURES OF PLASTER AND CHARCOAL.

Charcoal in Mixture		Potential Heat		Apparent Error in Measurement, Btu per lb
Dry Plaster Basis, per cent	Hydrated Plaster Basis, per cent	Computed, Btu per lb	Measured, Btu per lb	
0	0	−290[a]	−290	. . .
1	0.852	−170	−172	−2
2	1.705	−47	−29	+18
5	4.28	318	357	+39
10	8.64	934	833	−101
30	26.7	3 489	3 320	−169
50	45.4	6 142	6 140	−2
100	100.0	13 870[a]	13 870	. . .

[a] Computations were made on the assumption of exact specimen preparation and measured potential heat for charcoal and hydrated plaster of 13,870 Btu per lb and −290 Btu per lb respectively.

[b] Measured values are averages of two determinations with combustion promoter in concentrations of 50 per cent.

RESULTS

Table II presents the results of potential heat measurements made on a variety of building materials. The materials listed here were selected largely to provide an indication of the manner in which the test method would classify materials. It should not be assumed that all materials which conform to the brief descriptions given will show the same potential heat. Potential heat is tabulated here as heat release per unit weight, per unit volume, and per unit area for impermeable materials subject only to surface reactions. For discussion purposes, Table II also presents the flame spread index of those materials which have been tested by the radiant panel method (**12**).

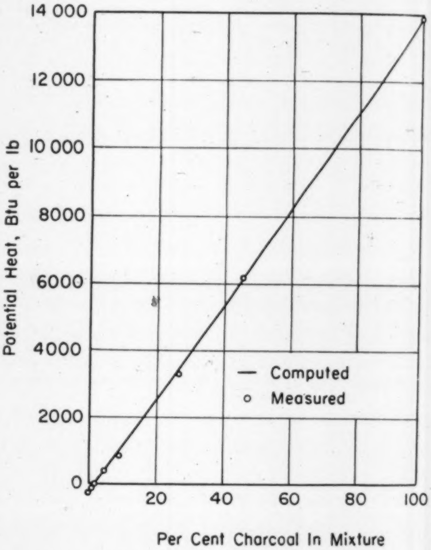

FIG. 3.—Comparison of Computed and Measured Potential Heat of Mixtures of Plaster and Charcoal.

Computations based upon measured potential heat for charcoal and hydrated plaster and assume exact specimen preparation.

Examination of this table shows that, for the materials studied, potential heat varies from about −300 Btu per lb to 17,000 Btu per lb. Materials such as brick, asbestos-cement board, concretes with the exception of cinder aggregate, and the gypsum plasters showed very low potential heat while cork, polystyrene, and asphalt-impregnated building paper showed very high values. Expanded metal lath, gypsum lath and wall board exhibited potential heat values which were in the range 300 to 1700 Btu per lb.

With materials such as woods and plastics, the quantity of residue (ash) remaining after firing was so small it was not necessary to make a calorimetric determination on the residue. In cases such as these, the potential heat reported was considered equivalent to the gross heat of combustion of the material.

The high potential heat of woods was not noticeably affected by impregnation with fire-retardant treatments for which studies were made. The apparent slight reduction of potential heat on a weight basis may be largely attributed to an increase in density resulting from the treatment process. Fire endurance tests of laminated panels (**13**) have shown only slight increase in the time to burn through treated panels as compared with that for untreated specimens. In one pair of panels of $2\frac{1}{2}$ in. over-all thickness built up with tongue and grooved core and $\frac{3}{16}$ in. veneer crossbanding, the times to flame-through were 72.1 and 69.2 min for retardant-treated and untreated specimens, respectively. However, other studies on built-up wood partitions of 4- and 6-in. thicknesses have shown up to 33 per cent improvement in fire endurance resulting from complete impregnation (**14**).

The fire retardant treatments did have a very significant effect upon the flame spread index values, also listed in Table II. The efficacy of the treatment appears to lie almost entirely in its ability to retard the surface spread of flame.

While one plastic, polystyrene, showed the highest potential heat measured during the study, two others showed potential heat similar to that of woods when compared on a weight basis.

The so-called noncombustible insulation materials tested exhibited potential heat which, while low, was not negligible. The potential heat for glass-fiber material resulted from its phenolic binder while that for mineral wool insulation included the contribution of the paper package enclosure. In the case of such low-density fibrous insulation materials, comparison of potential heat values may be more meaningful when considered on a volume basis.

The concretes in general exhibited low potential heat. The potential heat values for expanded slag, shale, and the siliceous aggregates were nearly zero. The potential heat for the concrete incorporating a calcareous aggregate was found to be slightly negative. This latter effect would be expected as a result of the heat absorption required for calcination of the aggregate and seems to imply that some calcination does take place during the brief high-temperature exposure within the bomb. The cinder aggregate concrete specimens tested exhibited significantly positive values of potential heat. The effect of the unburned fuel fraction in cinders contributing to the severity of building fires has been noted during burnout tests at the National Bureau of Standards.

Fire endurance tests on concrete masonry wall units performed by Menzel (**15**) have also shown that the addition of solid fuels to cinder aggregates reduced the gas requirement for performance of the test. This latter effect would, of course, be indicative of heat release by the specimen since control of the gas feed is such as to maintain a fixed time-temperature exposure relationship.

Due to endothermic chemical reactions, the gypsum plaster specimens

TABLE II.—POTENTIAL HEAT OF SELECTED BUILDING MATERIALS TOGETHER WITH FLAME SPREAD INDEX.

Material	Description, Dimensions and/or Composition	Thickness, in.	Density		Weight Loss at 105 C, per cent	Residue After Firing, per cent	Gross Heat of Combustion		Potential Heat			Flame Spread Index, I_s
			lb per cu ft	lb per sq ft[b]			Air Dry Material, Btu per lb	Residue After Firing, Btu per lb	Weight Basis, Btu per lb	Volume Basis, Btu per cu ft	Unit Area Basis, Btu per sq ft[b]	
1. *Woods*												
a. Douglas fir	Untreated	¾	38.0	...	8.3	0	8 400	[a]	8 400	319 × 10^3	...	69
b. Douglas fir	Fire retardant treatment "A"	¾	37.2	...	7.8	1.8	8 290	[a]	8 290	308	...	5
c. Douglas fir	Fire retardant treatment "B"	¾	47.2	...	9.9	1.6	7 860	[a]	7 860	371	...	3
d. Douglas fir	Fire retardant treatment "C"	¾	38.8	...	11.3	4.5	7 050	[a]	7 050	274	...	1
e. Maple, soft	Untreated	1	39.5	...	9.4	0.4	7 940	[a]	7 940	314	...	82
f. Hardboard	Untreated	¼	59.8	...	4.3	0.6	8 530	[a]	8 530	510	...	150
2. *Plastics*												
a. Polystyrene	Wall tile 8½ in. sq light green	0.075	65.4	...	0.1	2.3	17 420	[a]	17 420	1140	...	335
b. Rigid poly(vinyl chloride)	Retardant treated, dark gray	0.147	86.0	...	0.1	0	9 290	[a]	9 290	799	...	10
c. Phenolic laminate	Dark gray	0.063	76.4	...	3.1	1.4	7 740	[a]	7 740	592	...	107
d. Polycarbonate resin	Transparent amber	¼	78.7	...	0.1	0	13 330	[a]	13 330	1050	...	56
3. *Insulation*												
a. Glass fiber	Semi-rigid, no vapor barrier	1	3.0	...	1.6	59.4	3 020	−20	3 040	9.1	...	...
b. Mineral wool batting	Full thick—batting	3	2.4	...	0.7	91.4	1 210	160	1 050	2.5	...	...
c. Roof insulation board		1	10.4	...	1.8	65.9	3 350	−30	3 380	35.1	...	61
d. Cork	Reconstituted cork sheet	¼	14.8	...	6.9	0.4	11 110	[a]	11 110	164	...	174
e. Cellulose mineral board		2	47.8	...	9.8	48.4	2 280	30	2 250	108	...	1.3
4. *Concrete*												
a. Cinder aggregate	9% cement, 7% fly ash, 84% aggregate	...	93.0	...	2.3	94.0	3 260	180	3 080	286	...	0
b. Slag aggregate	8% cement, 9% pozzolan, 83% aggregate	...	110.1	...	0.9	94.8	40	−40	80	8.9	...	...
c. Shale aggregate	12% cement, 3% fly ash, 85% aggregate	...	80.5	...	0.9	95.3	−20	−30	10	0.5	...	...
d. Calcareous gravel aggregate	4% cement, 2% silica flour, 94% aggregate	...	133.1	...	0.4	82.2	−240	10	−250	−33.1	...	...
e. Siliceous gravel aggregate	12% cement, 39% sand, 49% aggregate	...	166.8	...	2.4	96.2	−70	−30	−40	−6.7	...	...
5. *Cement Board*												
a. Asbestos cement board	....	$^3/_{16}$	117.0	...	1.6	81.2	130	50	80	9.2	...	0
b. Asbestos cement board + 20 mil paint	....	$^3/_{16}$	159.2	...	1.7	79.0	400	10	390	62.4	...	0.7

3. *Gypsum*											
a. $CaSO_4 \cdot 2H_2O$	Hydrated neat gypsum—	0.41	137.9	...	19.0	79.9	−590	−300	−290	−31.3	...	0
b. Perlite aggregate plaster	79% plaster, 21% aggregate (1:2 ½ bags per cu ft)	1	53.2	...	12.8	84.1	−320	−390	70	4.0	...	0
c. Sand aggregate plaster	32% plaster, 68% aggregate (1:2 bags per cu ft)	1	101.8	...	6.6	92.1	−110	−60	−50	−5.4	...	0
d. Vermiculite aggregate plaster	85% plaster, 15% aggregate (1:2 bags per cu ft)	1	51.2	...	15.1	82.1	−460	−370	−90	−4.6	...	0
e. Gypsum board "A"	....	⅜	50.5	...	14.7	81.2	180	−580	760	38.4	...	14.3
f. Gypsum board "A" with paper removed	....	⅜	46.6	...	16.9	78.6	−720	−450	−270	−12.5	...	0
g. Gypsum board "A" + alkyd gloss paint	....	⅜	46.7	...	12.6	74.4	410	−470	880	41.2	...	8.0
h. Gypsum board "B"	....	½	51.2	...	17.9	73.6	100	−550	650	33.0	...	7.9
7. *Lath*											
a. Gypsum A	....	⅜	55.3	...	14.3	74.9	−20	−330	310	17.0	...	...
b. Metal diamond mesh	....	0.025	405	...	0	102.6	3 630	2 400	1 230	...	1370	0
c. Metal diamond mesh paint removed	....	0.019	401	...	0	102.6	3 060	2 400	660	...	420	0
8. *Metals*											
a. Structural steel—unpainted	8 by ½ in.	0.060	489	0.828	0	101.8	2 860	2 630	230	...	190	...
b. Magnesium	3 by ½ in.	0.128	122	0.500	0	105.4	11 080	280	10 800	...	5400	...
c. Aluminum	12 by 25/32 in.	0.004	165	0.025	0	101.0	13 010	12 980	30	...	0.8	...
d. Brass	10 by ¾ in.	0.004	534	0.089	0	102.2	1 380	1 280	100	...	8.9	...
e. Copper	6 by ½ in.	0.024	556	0.530	0	101.1	930	870	60	...	32	...
f. Lead	12 by ¾ in.	0.036	710	0.992	0	100.3	1 810	1 530	280	...	278	...
g. Zinc	0.470 in. diam by 0.819 in. long	...	415	3.16		100.1	2 950	2 190	760	...	2390	...
9. *Miscellaneous*											
a. Paint "E"	Dried paint film	0.05	...	...	...	0	3 640	[a]	3 640	...	...	30
b. Asphalt shingles	Fire retardant	¼	70.7	...	0.4	50.3	8 290	−30	8 320	588	...	123
c. Building paper	Asphalt impregnated	0.042	42.8	...	3.9	2.1	13 620	[a]	13 620	583	...	385
d. Building paper	Rosin sized	0.018	23.6	...	6.0	0	7 650	[a]	7 650	181	...	192
e. Linoleum tile	Laminated tile 9 in. sq marbleized	⅛	86.0	—	1.5	45.4	7 720	−40	7 760	667	...	106
f. Brick red-face	....	2¼	139.1	...	0.1	99.7	60	40	20	2.2	...	0
g. Charcoal	Activated coconut charcoal, 6–14 mesh	...	...	...	...	0	13 870	[a]	13 870	...	...	...

All weights and percentages refer to original air-dry weight.

[a] Potential heat was assumed equal to the gross heating value of the raw material because of the low residue after firing.

[b] Based upon total exposed surface area.

showed low or negative potential heat. The potential heat of pure hydrated neat gypsum plaster was found to be −290 Btu per lb. Higher potential heat was obtained with the paper-coated gypsum wallboard specimens than with the plasters due to the contribution of the paper facing. A slightly higher potential heat was obtained with a gypsum wallboard specimen containing a paint overcoat.

The laths studied included both gypsum lath and expanded metal lath. The potential heat of the gypsum lath was slightly less than that of gypsum wall boards of the same thickness. That for the metal lath was largely due to the paint which had been applied and the surface oxidation of the metal.

In preparing metal specimens for firing, sheet strips were multiply-folded to the dimensions required. Of the metals tested, only steel, copper, and brass did not melt at 750 C, and since these were subject to surface oxidation, it seemed proper to report potential heat on the basis of the exposed surface area. Magnesium was the only metal tested which burned when exposed to the conditions of the muffle furnace. With the exception of magnesium, the oxidation of the metals studied was not severe and potential heat was low but not zero.

DISCUSSION

Experience with the test procedure indicates that it is applicable to a wide variety of building materials. It appears to provide a useful tool for determining potential heat which is a measure of that portion of the gross heat of combustion liberated during exposure to a simulated standard fire at 750 C. The method has the advantage over previously used combustibility test methods of providing a quantitative measurement of the heat release during such exposure and is particularly suited for measurements on metals and on materials of low combustibility.

The data presented in Table II show little correlation between the potential heat and flame spread index properties of the materials studied. This fact raises questions about the usefulness of surface flammability test methods for assessing the heat release likely to be experienced from building materials during fires.

The only other calorimetric method of a similar nature known to be used in this country at the present time is the Factory Mutual Laboratories' Construction Materials Calorimeter (8). This method requires a large specimen 4 by 4 ft in size and representative in thickness and construction of the building structure to be simulated. It provides a useful means for measuring the rate at which the structure will liberate heat during fire exposure on one surface. Also, by integration techniques, it can provide an indication of the heat release rates for fires of various durations. However, because of the problems associated with heat losses through the specimen, it seems doubtful that this method is suitable for total heat release measurements. It seems likely that for the case of very thin panels of translucent combustible materials, such as plastics, such losses could very well exceed the heat of combustion.

Probably the two methods should be considered as complementary, one providing a laboratory method for measurement of total heat release with small specimens and the other providing for measurement of the rate at which heat generation by the large specimen exceeds the losses through it. Certainly, studies performed with the use of this large-scale test method would prove useful in showing the appropriate manner to be used in application of the laboratory test results.

During the studies performed, only one material, graphite, failed to burn in the bomb but was consumed in the firing process. Complete combustion of graphite in the bomb was not obtained even with the addition of an equal weight of combustion promoter. The problems of calorimetric measurements with graphite have been reported (16). Accordingly, when the presence of graphite is suspected, the residue from the bomb should be examined and, if necessary, fired to determine the amount of unconsumed carbon by weight-loss measurements. Charcoal which comprises carbon with some chemically bonded hydrogen was not subject to this difficulty and was readily burned in the bomb. For all the materials tested, corrections for the nitrogen oxidation amounted to less than 100 Btu per lb. Except in the case of gypsum, no sulfur corrections were necessary.

TABLE III.—ESTIMATED CORRECTIONS TO POTENTIAL HEAT MEASUREMENTS FOR HEAT OF VAPORIZATION EFFECT.[a]

Material	Water Present and Resulting from Combustion, per cent by weight	Potential Heat Correction, Btu per lb
Douglas fir	64	−1130
Styrene	69	−1180
Vinyl chloride	43	−740
Gypsum plaster (neat)	21	−360
Concrete	6	−102

[a] Based on enthalpy difference at atmospheric pressure between that of water at 20 C and steam at 750 C.

Mention should be made of the fact that the calorimetric methods used result in the condensation of water formed as a product of combustion. Because of this, the values reported are representative of the gross heat of combustion of the materials and, as such, include the heat liberated on condensation of the water vapor. During fire exposure, however, the bulk of the water vapor formed escapes without condensation and the net heat of combustion probably gives a closer approximation to the heat available. It appears beyond the scope of this paper to present a complete discussion of this aspect of the procedure. However, the data in Table III are presented for the purpose of estimating the magnitude of this effect. The data presented were determined from steam table data for the difference in enthalpy between water at 20 C and steam at 750 C, both at atmospheric pressure. It was further assumed that the combined and free water initially present in the material were not chemically changed. Actually this latter assumption appears likely to be proper for all materials listed with the single exception of gypsum. The negative potential heat listed in Table II for gypsum appears to result from at least partial chemical decomposition of the hydrated gypsum. For the other materials listed the applicable correction for water is but a small fraction of the value of the potential heat. Table III is presented here only for discussion purposes. It appears inadvisable to attempt correction of experimental potential heat data for the purpose of compensation of these heat of vaporization effects.

CONCLUSION

The following conclusions seem justified on the basis of the work reported:

1. A method has been developed for measuring the potential heat of building materials with the objective of providing an indication of the heat release likely to occur during building fires.

2. Measurements have been made of both potential heat and flame spread index properties of a wide variety of building materials. In general, there was little correlation observed between these two material properties.

3. For gypsum, chemical decomposition reactions result in a potential heat for this material which appears to partially account for endothermic effects during fire exposure. This effect was also

observed for two concretes but was not observed for other materials in which potential heat values measured should be considered indicative of the combined heat of combustion and heat of condensation of water vapor.

APPENDIX

EXPERIMENTAL PROCEDURE

This procedure is suitable for determinations on simple materials or on composite assemblies made up of a number of materials which can be pulverized into a homogeneous mixture.[4]

Specimen.—Two air-dry test samples are required for each determination, both must be representative of the material or assembly involved. A sample was considered air-dry when in moisture equilibrium with an atmosphere maintained at 73 ± 5 F and 50 ± 5 per cent relative humidity. Treatment of these two samples are described separately:

Specimen for Direct Bomb Test:

1. All or a representative portion of this specimen was pulverized into a form suitable to pass a No. 60 sieve.
2. A 1-g pellet of a representative sample of the powder formed in step 1 was prepared.
3. This pellet was used as the test specimen following the procedures for determination of the heat of combustion recommended in reference (**10**). The usual sulfur and acid corrections were made. These take into account the oxidation of sulfur and nitrogen, if present, which would not normally occur during fire exposure.
4. The fuel capsule was examined after firing the bomb. If the pellet burned completely, leaving no significant amount of ash, the heat of combustion computed on an air-dry basis was recorded and steps 5 through 7 were omitted.
5. If the pellet did not burn or a significant amount of ash remained after step 4, a second 1-g pellet was prepared with an intimate mixture of the powdered sample and a standard sample of benzoic acid in equal weight proportions.
6. The pellet prepared under step 5 was used as the test specimen following the same procedures as for item 3 above.
7. A correction for the heat of combustion of the benzoic acid present in the pellet was applied to the measured heat released by the specimen and the heat of combustion was then computed on an air-dry basis.

Specimen for Muffle and Bomb Test:

1. A representative air-dry specimen of the material, or assembly, was cut in the form of a rectangular prism having dimensions of ½ by ¾ by 3 in. In the case of sheet materials, folded or laminated assemblies were formed to these dimensions.
2. The weighed specimen was supported in a fused silica container of 1¼ in. inside diameter by 4-in. length. The specimen, container cap, and a tube for supply of air to the bottom of the container were assembled. The assembly was then placed on a firebrick support within an electric muffle furnace maintained at 750 C. Air was supplied at a rate of 0.1 cu ft per min measured under laboratory conditions to assist in oxidation of the specimen. In cases where ignition occurred immediately, application of air was delayed until initial flaming had stopped. Firing of the specimen at the above described rate of air supply was continued for 2 hr.
3. The silica container with specimen was removed and placed in a desiccator to cool. Measurements were then made to determine the weight loss of the specimen.
4. If the residue from the firing procedure

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[4] Caution should be observed when performing bomb calorimetric measurements on materials containing significant proportions of metallic ingredients. Apart from the high reaction temperatures which may occur with the resulting possible involvement of portions of the bomb, consideration should be given to the possibility of electrical shorts in the ignition system.

was less than 5 per cent of the initial specimen weight, the following steps were omitted and the heat of combustion determined under the direct bomb test was reported as the potential heat of the material.

5. If the residue from step 4 was in excess of 5 per cent of the specimen weight, this residue was pulverized and the same procedures used as for steps 5 through 7, under the direct bomb test. The resulting heat of combustion was reported as that of the residue.

6. The heat of combustion of the residue was multiplied by the ratio of the residue weight to the original specimen weight.

7. The resulting difference in the heats of combustion was a measure of the heat released during the firing process in the muffle furnace and was reported as the potential heat.

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DISCUSSION

MR. L. BOOR.[1]—The topic discussed was one of great interest in the early days of plastics, most of which were highly flammable. The same sort of calorimeter approach was tried, but one of the aspects of this kind of testing became what has been referred to as "vigorously controversial." That is, the heat of evaporation of the water in open flame is lost, and in the calorimeter it is retained.

Is there any way of getting around that or how do you resolve that?

MR. NOLAN D. MITCHELL.[2]—I want to compliment the authors on the development of this process. They do not, however, cover the question of laminated materials which have highly combustible materials exposed to fire and rapid combustion, as facings over other materials which do not burn so rapidly. The procedure gives us only the total heat released upon complete combustion. It occurs to me that rate of heat release is just as important as having the total heat released.

Another thing mentioned is the great difference in the treated wood and the untreated wood, as far as flame spread is concerned, but as a matter of fact when you have conditions to cause the treated wood to burn, you get fairly close to the total heat release that you would have with the untreated wood. That has been observed in our fire tests at the Bureau of Standards.

MESSRS. J. J. LOFTUS, D. GROSS, AND A. F. ROBERTSON (*authors' closure*).—The authors appreciate the comments of Messrs. Boor and Mitchell.

The question raised by Mr. Boor is one for which no specific answer can be given since the quantity of heat represented by the evaporation of water depends upon assumptions applicable to the particular fire situation. The assumptions used in estimating the corrections in Table III, namely complete combustion, steam superheated to 750 C and no recondensation, were illustrative and represent only one set of conditions. We have followed the usual practice in making heat balance calculations by reporting the gross values and suggesting in this table a method of correcting for the heat content of water vapor for these conditions.

The importance of the rate of heat release of materials in building fires was emphasized by Mr. Mitchell. In addition to the total heat which any building element may contribute to support a fire, the rapidity with which heat is developed is obviously of significance and thus calorimeters such as that used by the Factory Mutual Laboratories seem appropriate for such measurements. From a regulatory standpoint, it seems reasonable that consideration might be given to the establishment of safe limits for the potential heat, rate of heat release and surface flammability of materials for particular occupancy requirements.

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[1] Chief-Instrumentation, Military Clothing and Textile Supply Agency, Technical Division, Philadelphia Quartermaster Depot, Philadelphia, Pa.

[2] Formerly of the National Bureau of Standards; Arlington, Va.

A METHOD FOR DETERMINING THE PLASTIC FLOW PROPERTIES OF SHEET AND ROUND TENSION TEST SPECIMENS*

By J. Nunes[1] and F. R. Larson[1]

Synopsis

An experimental procedure is described for obtaining continuous load-profile measurements of sheet and round tension specimens during plastic flow. Some data obtained on tempered alloy steel, titanium, 301 stainless steel and an aluminum alloy are presented to illustrate the uses and advantages of this testing method. With this procedure, it was possible to obtain the following data from a single test curve; true stress-strain, true strain rate, mechanical anisotropy and "corrected" flow stress.

Tests were conducted primarily at room temperature on sheet material with some low-temperature tests conducted on the 301 stainless steel. A round tension specimen of aluminum was also tested to illustrate a method for determining "corrected" flow stress values according to the Bridgman technique.

Ludwik (**1**)[2] was the first to use true stress-strain data in plastic flow analysis. These data were obtained by measuring the instantaneous minimum diameter of the specimen with a micrometer at some corresponding instantaneous load. MacGregor (**2**) was perhaps the principal exponent of this type of test for the useful analysis of plastic flow properties. Interest in obtaining more basic plastic flow properties from the simple tension test has led later investigators (**3-7**) into devising methods for obtaining true stress-strain data more effectively. These devices were mechanical (**3,4**) or electromechanical (**5,6**) diameter gages which were usually manually operated. Recent developments have improved this type of gage to the point where it is now possible to obtain minimum diameter readings automatically at extremely low testing temperatures (**7**). However, as all these gages have been employed in determining the minimum diameter of a round tension specimen and were not suitable for sheet specimens, it was felt that a method for measuring sheet tension specimens should be developed. Also, such tests would be of value because of the general lack of true stress-strain data available on sheet and the tremendous amount of interest currently centered around high-strength sheet materials.

It is the purpose of this paper to describe the test method developed and to illustrate the application of this method for determining such plastic flow properties as true stress-strain, true strain rate, mechanical anisotropy, and "corrected" flow stress.

Most of the data used in this investigation have been obtained from a previous study on high-strength sheet materials (8). Data were obtained on a round tension specimen of aluminum in order to illustrate a method for determining "corrected" flow stress values from neck geometry measurements (9). All tests were conducted at room temperature with the exception of the 301 stainless steel which was tested over a temperature range of room temperature to −196 C.

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* Presented at the Sixty-fourth Annual Meeting of the Society, June 25–30, 1961.

[1] Materials Engineer and Physical Metallurgist, respectively, U. S. Department of the Army, Ordnance Corps, Watertown Arsenal Laboratories, Watertown, Mass.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

DESCRIPTION AND OPERATION OF APPARATUS

Powell, Marshall, and Backofen (6) designed a diameter gage whose basic features are maintained in the gage used here. However, the most important modification in the present gage was the addition of a pair of cantilever arms 90 deg opposite the originally designed pair. This was first done at Watertown Arsenal Laboratories several years ago in order to obtain average area measurements of the elliptical cross-sections encountered in tension tests of round titanium specimens. By changing the Wheatstone circuit of this gage to give independent signals of width and thickness change and by modifying the cantilever arms in the width direction, it was possible to accommodate sheet specimens.

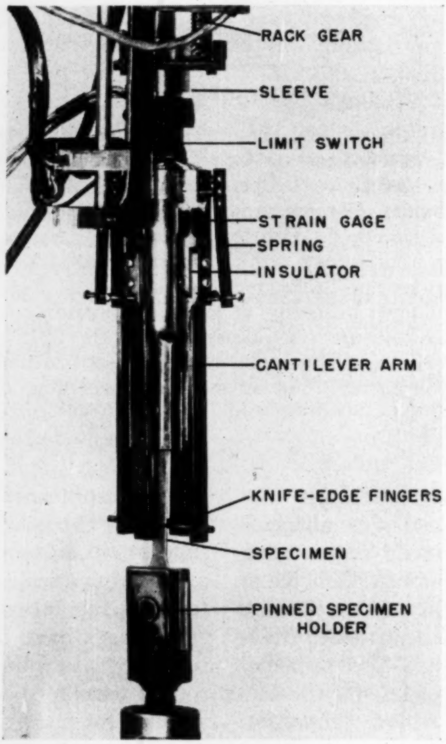

FIG. 1.—Diameter Gage Assembly.

Basic components of the diameter gage assembly are shown in Fig. 1 with a sample specimen in testing position. Located on the lower end of the cantilever arms are knife-edge fingers which are adjustable to the specimen cross-section. These fingers act as sensing elements by deflecting when the specimen deforms. These deflections are converted to electrical signals at the strain gages located at the upper end of the cantilever arms. Placed between the strain gage component and the cantilever arm is an insulator used to minimize the temperature gradient effects obtained at low testing temperatures. An epoxy resin adhesive was used to bond the strain gages on the sides of the cantilever arm assembly. A spring is attached, as shown, to each arm to reduce any vibration picked up by the arm as it traverses the specimen surface. The sleeve, on which the cantilever arm assembly is attached, was made to align with the shaft holding the tension specimen and was free to slide vertically along the shaft. By attaching a rack gear and a motor to the assembly, it was possible to obtain continuous travel along the specimen surface at a predetermined rate set by a Variac control. The limit switches were provided to control the total travel of the diameter gage assembly in either direction and are adjustable to the specimen gage length.

Illustrated in Fig. 2 is the wiring diagram of the Wheatstone Bridge circuit employed in this assembly. The C_1 and C_2 resistors are placed on the compressive side of the cantilever arms with the T_1 and T_2 resistors placed on the tension side as shown. This circuit is the same for both pairs of arms, which results in signals being obtained independently in the width and thickness directions of a specimen.

A photograph of the test setup is shown in Fig. 3 with a specimen in position ready for testing. It can be seen that

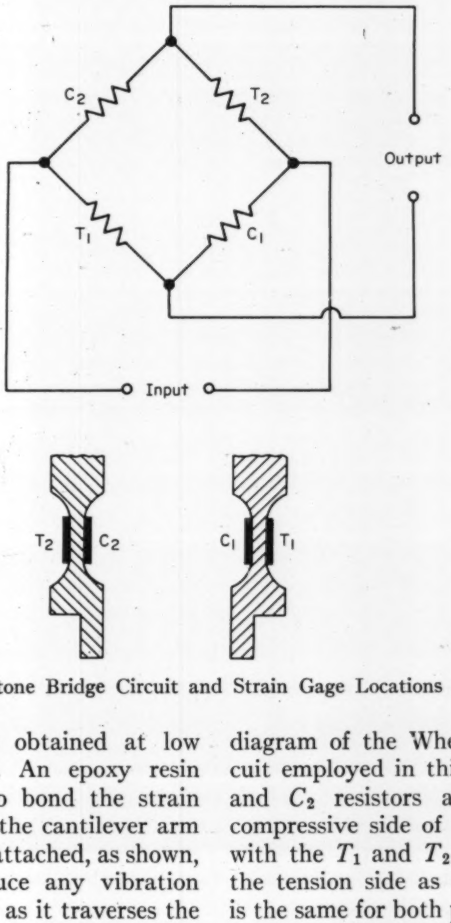

FIG. 2.—Wheatstone Bridge Circuit and Strain Gage Locations on Cantilever.

the diameter gage assembly is relatively compact and is positioned over the specimen in such a manner that a liquid coolant tank can be placed into the setup for low-temperature testing. The test machine partially shown here is a 30,000-lb capacity mechanical testing machine. Two shafts mounted between the platen heads of the machine hold the specimen. One of these is simply a pinned holder, while the other is made up of two parts, a holder and a dynamometer through which the load was recorded. To the left is a six-channel oscillographic recorder which was used to obtain a record of the load-profile measurements.

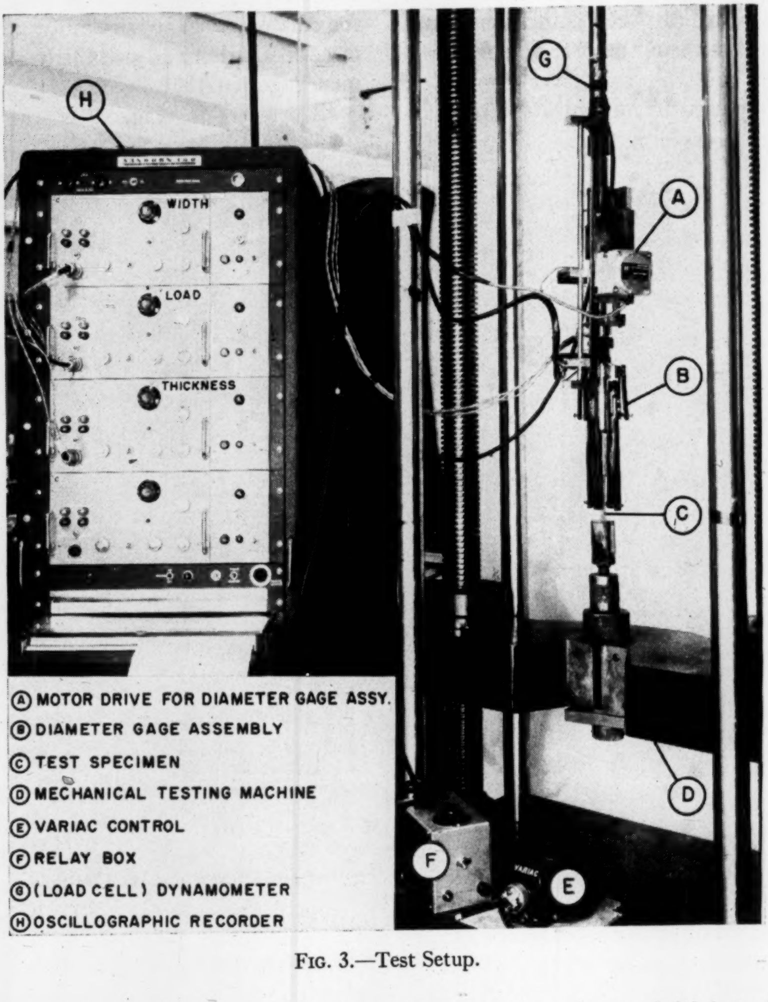
G
H
A
B
C
D
F
E
VARIAC
WIDTH
LOAD
THICKNESS
Ⓐ MOTOR DRIVE FOR DIAMETER GAGE ASSY.
Ⓑ DIAMETER GAGE ASSEMBLY
Ⓒ TEST SPECIMEN
Ⓓ MECHANICAL TESTING MACHINE
Ⓔ VARIAC CONTROL
Ⓕ RELAY BOX
Ⓖ (LOAD CELL) DYNAMOMETER
Ⓗ OSCILLOGRAPHIC RECORDER

FIG. 3.—Test Setup.

All tests were run at a constant crosshead speed to give an initial strain rate of 0.01 per min. Records of load versus crosshead displacement were used to obtain yield strength values at the lower testing temperatures. Tests conducted at room temperature were run with a snap-on extensometer attached to the specimen to 0.2 per cent offset yield with the diameter gage retracted in the "up" position. After the desired yield was obtained, the diameter gage was set into operation to record the resulting deformation to fracture.

Figure 4 shows a typical record. Only selected segments are shown as it would have been impractical to reproduce the entire chart length here. Representative points are taken at different time intervals to serve as an example of the type of curve obtained. Each segment shown has two complete cycles of width or thickness. These cycles are mirror images (at slow rates of deformation) as they represent the profile records obtained on the upward and the downward scan. One scan or cycle represents 2.75 in. gage length for the sheet specimens and 1.25 in. gage length for the round specimens.

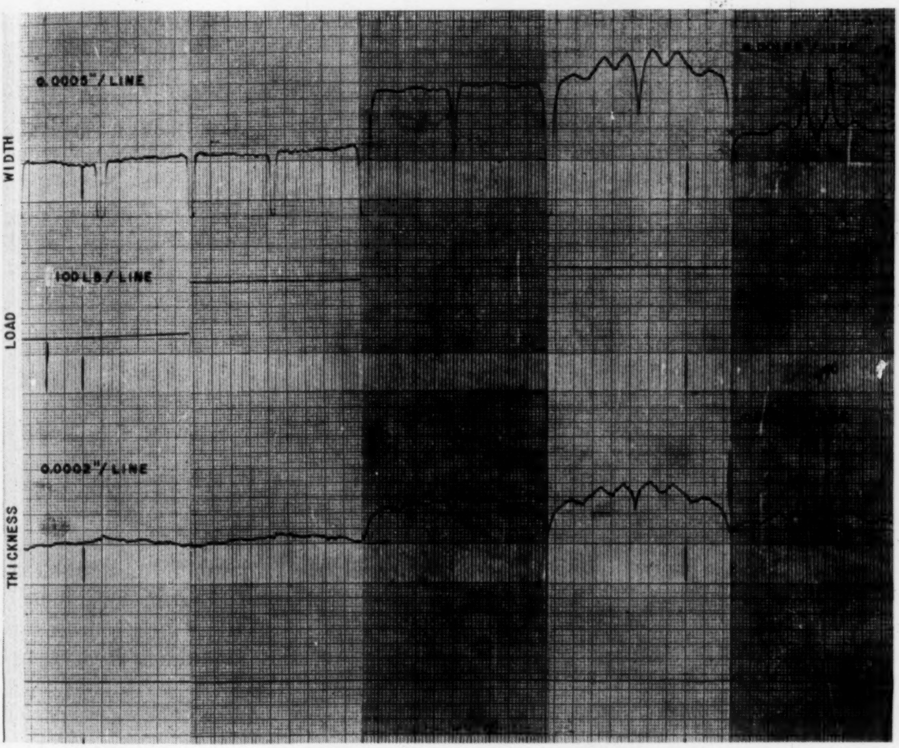

FIG. 4.—Typical Load-Profile Record.

TRUE STRESS-STRAIN DETERMINATIONS

To orientate the reader, the room temperature plastic flow properties and the yield stress and ultimate tensile stress for all the materials mentioned are listed in Table I along with the dimensions of the type of specimens used. A more complete analysis of the sheet materials studied can be found in the Larson and Nunes paper (8).

By definition, true stress, σ, and true strain, ϵ, are:

$$\sigma = \frac{P}{A_i} \quad \text{and} \quad \epsilon = \ln \frac{A_0}{A_i} \quad \ldots\ldots(1)$$

where:

P = applied load,

TABLE I.—MECHANICAL PROPERTIES AT ROOM TEMPERATURE.

Material	Width, in.	Thickness, in.	$\sigma_{0.04}$[b], psi	σ_{ml}[b], psi	σ_f[b], psi	ϵ_{ml}[b]	ϵ_{f-t}[b]	n[b]	Yield Stress, 0.2%, psi	Ultimate Tensile Stress, psi
Steel										
H-11—950 F temper...	0.372	0.127	296 200	305 400	371 900	0.060	0.243	0.062	217 000	287 200
1000 F temper...	0.372	0.127	286 900	281 300	355 300	0.024	0.235	0.060	216 000	274 700
1050 F temper...	0.372	0.127	266 700	262 600	354 600	0.029	0.346	0.053	212 800	255 300
1100 F temper...	0.372	0.127	237 000	243 700	311 000	0.059	0.328	0.058	195 700	229 800
1200 F temper...	0.372	0.127	151 500	159 400	229 700	0.068	0.434	0.074	120 200	148 900
1300 F temper...	0.372	0.127	127 500	141 200	205 000	0.098	0.434	0.102	119 000	127 900
301 stainless—¾ hard..	0.367	0.108	151 000	235 000	329 000	0.348	0.463	0.330	131 300	159 100
AMS 5548[a] 900 F age...	0.272	0.057	198 000	220 500	283 400	0.118	0.324	0.136	184 000	195 600
1000 F age...	0.272	0.057	170 000	193 000	255 300	0.112	0.428	0.120	155 000	172 500
Titanium										
16V-2.5Al............	0.500	0.056	122 500	134 400	145 300	0.100	0.173	0.098	105 700	118 600
4Al-3Mo-1V..........	0.500	0.059	101 000	103 600	150 300	0.051	0.107	0.074	98 600	99 700
Aluminum										
24S-T4..............	0.252-diam		58 500	79 100	100 000	0.148	0.442	0.212	44 000	68 300

[a] Aeronautical Materials Specification. Semi-austenitic stainless steel with chromium-nickel-molybdenum and no aluminum.

[b] Symbol nomenclature—$\sigma_{0.04}$ = True stress at $\epsilon = 0.04$
σ_{ml} = True stress at maximum load
σ_f = True stress at fracture
ϵ_{ml} = True strain maximum load
ϵ_{f-t} = True strain in the thickness direction at fracture
n = Strain hardening exponent

A_o = original area, and
A_i = instantaneous area.

For round bars the true strain (based on diameter measurements) would simply be:

$$\epsilon = 2 \ln \frac{D_0}{D_i} \qquad (2)$$

Also as $\epsilon = \epsilon_w + \epsilon_t$, the width and thickness strains, ϵ_w and ϵ_t respectively, may be expressed as:

$$\epsilon_w = \ln \frac{W_0}{W_i} \quad \text{and} \quad \epsilon_t = \ln \frac{T_0}{T_i} \qquad (3)$$

The width and thickness were measured independently from the load-profile record. These values were taken at the minimum cross-section with load readings taken at the corresponding point.

The true stress-strain data obtained in this manner for three types of sheet material, H-11, titanium, and 301 stainless steel are illustrated in Figs. 5 to 7. It can be seen that these curves are similar to those obtained from round tension specimens.

For determining the work-hardening property from this type of test, the data can be replotted on log-log paper. The slope of this plot, n, is from the equation:

$$\sigma = s_1 \epsilon^n \qquad (4)$$

This exponent is called the strain-hardening exponent and represents the work-hardening ability of the material; S_1 is the intercept of the flow stress at a strain of 1. Low values indicate low rates of work hardening. Figure 8 illustrates a curve of this type for the H-11 steel. As no stress corrections are made for the hydrostatic stress component which may have been introduced in the flow stress values observed due to necking, the constant slope, *n*, does not go through the remaining points after maximum load.

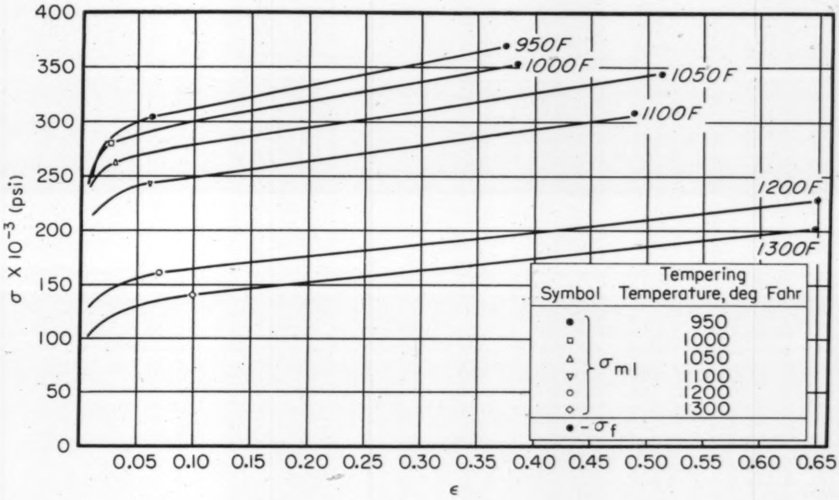

FIG. 5.—Room Temperature True Stress-Strain Curves of H-11 at Various Strength Levels.

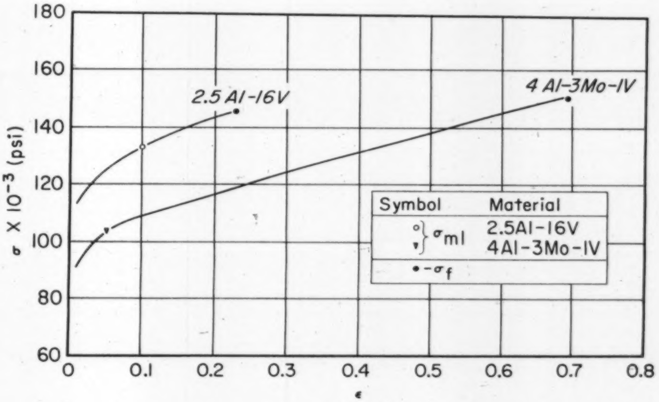

FIG. 6.—Room Temperature True Stress-Strain Curves for Titanium 16V-2.5Al and 4Al-3Mo-1V.

TRUE STRAIN RATE DETERMINATION

As a more continuous curve is obtained with the electro-mechanical diameter gage (by recording at a constant chart speed and by measuring the specimen at a constant rate), it was possible in this investigation to see the development of local deformation and to determine the true strain rate at any point along the specimen. True strain rate by definition is:

$$\dot{\epsilon} = \frac{d\epsilon}{dt} \qquad (5)$$

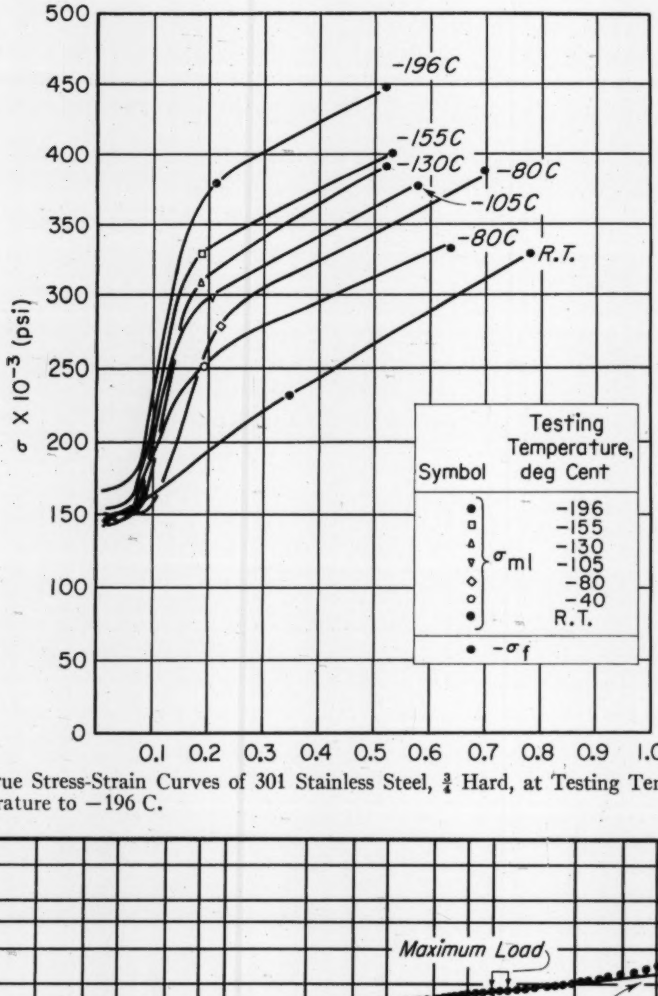

FIG. 7.—True Stress-Strain Curves of 301 Stainless Steel, ¾ Hard, at Testing Temperatures of Room Temperature to −196 C.

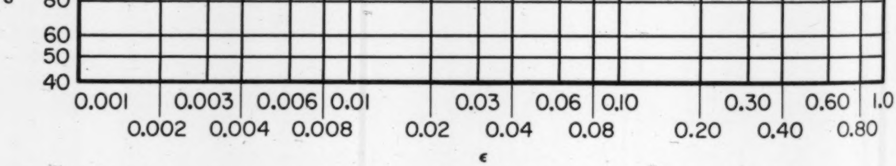

FIG. 8.—Room Temperature Log True Stress Versus Log True Strain Curve for H-11 at 1300 F Temper.

It was felt that a description of the variation of true strain rate at the minimum cross-section would be of interest. Figure 9 dramatically illustrates this true strain rate variation with the true strain. Superimposed on this plot are the curves for constant true strain rate and the true strain rate obtained during uniform deformation (at constant crosshead speed). It can be seen that strain rate determinations based upon a constant crosshead speed can be considerably in error once local deformation ensues. These true strain rate data were determined graphically from strain-time plots similar to that shown in Fig. 10 by measuring the slope at various points along the curve.

The material shown in Fig. 10 is $\frac{3}{4}$ hard 301 stainless steel. Variations of the strain rate can be seen to be occurring. The corresponding profiles are shown to illustrate how this curve is related to the profile record.

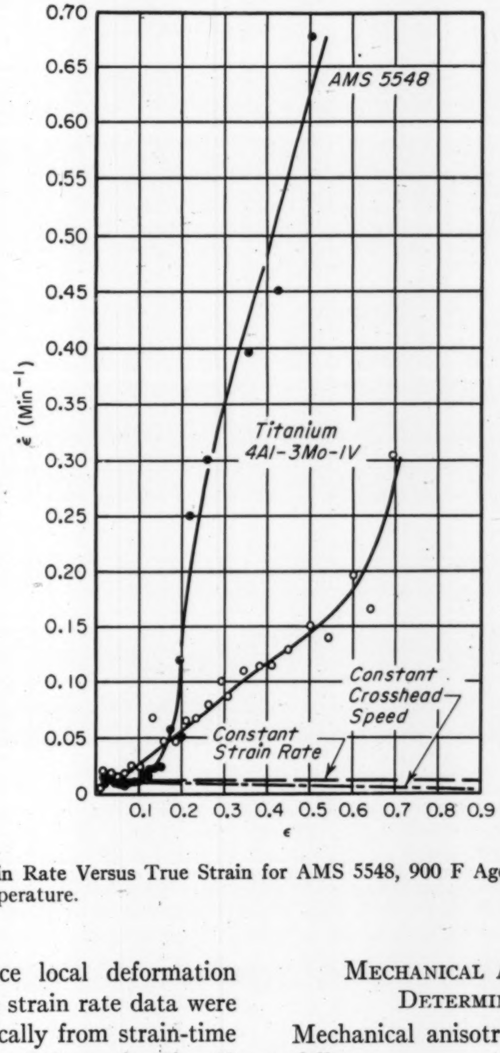

FIG. 9.—True Strain Rate Versus True Strain for AMS 5548, 900 F Age and Ti-4Al-3Mo-1V, Tested at Room Temperature.

MECHANICAL ANISOTROPY DETERMINATION

Mechanical anisotropy, K, is defined as follows:

$$K = \epsilon_W/\epsilon_t \quad \ldots\ldots\ldots\ldots (6)$$

As $\epsilon = \epsilon_w + \epsilon_t$, by substituting, Eq 6 becomes:

$$K = \epsilon/\epsilon_t - 1 \quad \ldots\ldots\ldots (7)$$

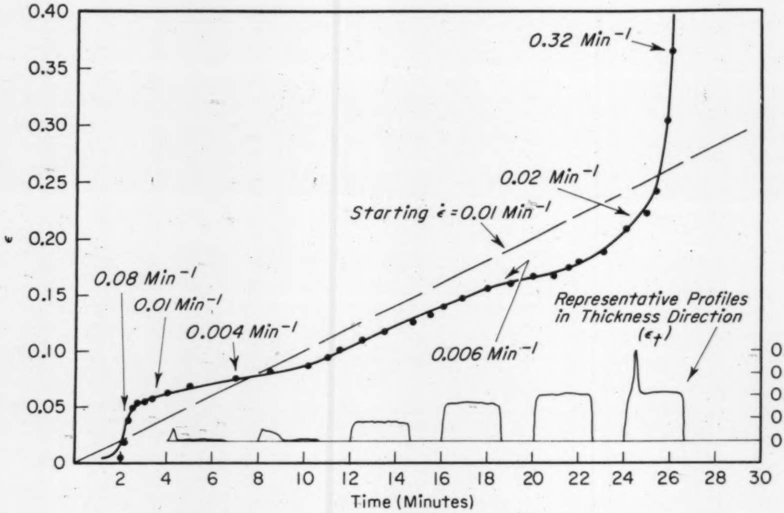

FIG. 10.—True Strain Versus Time Plot for 301 Stainless Steel at −196 C.

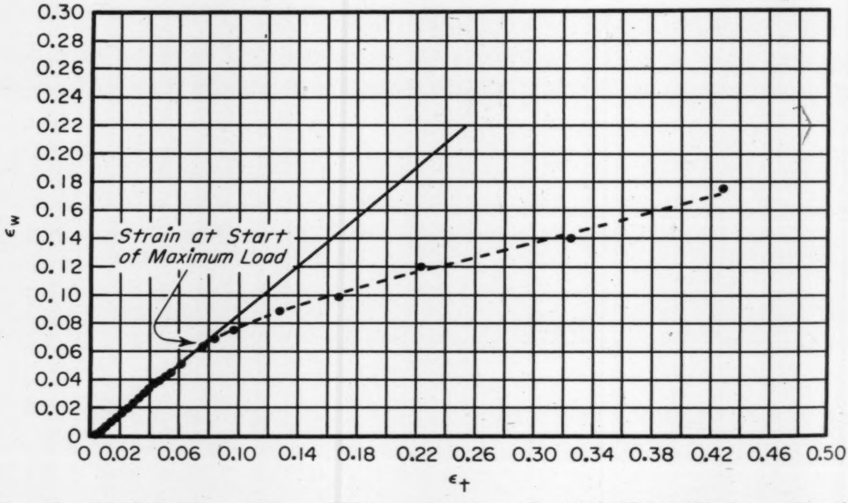

FIG. 11.—Width Strain, ϵ_w, Versus Thickness Strain, ϵ_t, for AMS 5548, 1000 F Age, Tested at Room Temperature.

Experimentally, *K* was determined by plotting ϵ_w versus ϵ_t and measuring the slope of the linear portion of the resulting curve. Figure 11 shows a plot of this type. It can be seen that nonlinearity occurs approximately after the strain at maximum load. This departure from linearity can probably be attributed to the fact that a shear band forms in the thickness direction which localizes the width strain to the width of the band, resulting in lower measured values of ϵ_w

than would have perhaps been observed if no band had occurred. This apparently was the case for the titanium tested, where no shear bands were observed and where it was found that the curves remained essentially linear to the vicinity of fracture.

TABLE II.—MECHANICAL ANISOTROPY VALUES, K, FOR THE VARIOUS MATERIALS TESTED AT ROOM TEMPERATURE.

Steel	Titanium	K
	4Al-3Mo-1V..........	7.20
H-11..............................		0.95[a]
301 SS..............................		0.94
AMS 5548..............................		0.93[a]
	2.5Al-16V..........	0.46

[a] Average values taken from more than one test.

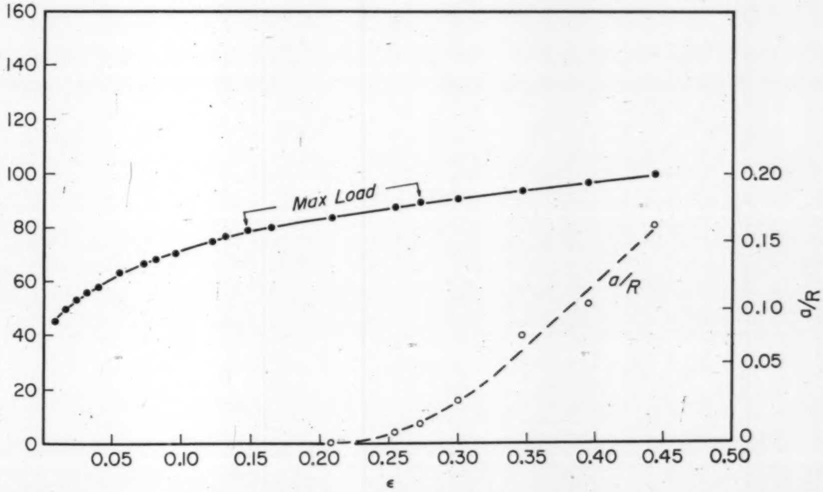

FIG. 12.—Room Temperature True Stress-Strain and a/R Curves for 24S-T4 Aluminum.

Anisotropy measurements in the plane undergoing maximum deformation (the minimum cross-section) are of interest as they tend to show the relative resistance to deformation of one principal strain direction to another. This may be extremely important if it can be shown that such phenomenon as fracture is dependent on the anisotropic behavior of a material. Tabulated in Table II are values of K obtained for the sheet materials mentioned. The K value of 7.2 for the titanium material shown demonstrates a relatively strong effect of directionality. This type of behavior has been known to occur for titanium due to crystallographic effects creating preferred planes of deformation.

"CORRECTED" FLOW STRESS DETERMINATION

Referring to Fig. 8, it can be seen that after local necking occurs, the experimental stress points do not follow the slope, n. This phenomenon may be attributed to the local stress increasing due to the formation of hydrostatic stress components at the neck. Bridgman (9) has formulated an expression for correcting this stress based on the above assumption. This "corrected" flow stress, σ_t, has been shown to follow the slope n to fracture and has been generally accepted as a reasonable description of the flow stress existing if no neck were present. Marshall and Shaw (**10**) measured the neck geometry of a steel and copper tension specimen and were able to calculate the σ_t values from Bridgman's equation given below:

$$\sigma_t = \frac{\sigma}{(1 + 2R/a) \ln (1 + a/2R)} \quad \ldots (8)$$

where:

a = radius at the minimum cross-section and

R = radius of curvature at the neck.

TABLE III.—COMPARISON OF TRUE STRESS DURING NECKING WITH CALCULATED AND "CORRECTED" FLOW STRESSES FOR 24S-T4 ALUMINUM.

True Strain, ϵ	a/R	True Stress, σ, psi	"Corrected" Stress, σ_t, psi	Calculated Stress, σ_n,[a] psi
0.208.....	0.002	84 000	83 900	83 500
0.254.....	0.009	87 900	87 500	86 900
0.272.....	0.015	89 500	88 800	88 100
0.299.....	0.032	90 500	89 100	89 700
0.346.....	0.080	93 500	91 700	92 500
0.394.....	0.103	96 400	94 400	94 800
0.442.....	0.162	100 000	96 000	96 800

[a] Based on n from $\sigma = S_1\epsilon^n$
$n = 0.196$
$S_1 = 114,000$

R was measured by Marshall and Shaw (**10**) by placing a metal cone against the neck and reading off the radius at a point where the cone and neck matched. To see if the method for obtaining a specimen profile used in this investigation could be employed toward determining the radius of curvature at the neck, an aluminum alloy was tested at room temperature. Because the correction of σ to σ_t is much simpler with a round isotropic tension specimen, this type was used. Figure 12 illustrates the true stress-strain curve for this material and the ratio of a to R, which was calculated from the following formula:[3]

$$a/R = \frac{a_1 D_i}{L_1^2 + a_1^2} \quad \ldots\ldots\ldots\ldots (9)$$

where:

$2L_1$ = cord of circle of radius R,

a_1 = perpendicular distance from the center of the cord to the center of its arc, and

D_i = instantaneous diameter ($2a$).

Listed in Table III are the true stress-strain values and the corresponding corrected, σ_t, and theoretical, σ_n, flow stresses which were calculated from Eq 5. It can be seen that the σ_t and σ_n values are in close agreement. Also tabulated are the a/R values which were obtained and used in calculating σ_t.

CONCLUSIONS

Specifically, the method used here has been shown to be capable of obtaining relatively continuous load-profile records of both sheet and round tension specimens at moderate rates of strain and test temperatures down to −196 C. It has been demonstrated that basic plastic flow properties can be adequately and relatively easily obtained for the materials illustrated here (from the simple tension test.)

Acknowledgment:

The authors wish to acknowledge the assistance given by Thomas S. DeSisto of Watertown Arsenal Laboratories, who has been primarily instrumental in the development of diameter gages and measuring devices similar to that employed by the authors in this paper.

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[3] Derivation of Eq 9 is found in the Appendix

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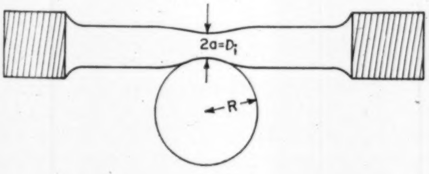
APPENDIX

DETERMINATION OF a/R FROM NECKING GEOMETRY

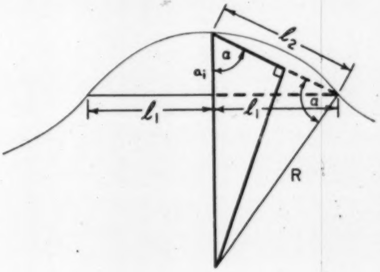


a = Radius of specimen at minimum section and

R = Radius of curvature at minimum section.

Constructing the following geometrical figure:

a_1 = Perpendicular distance from arc to center of chord and



$2l_1$ = Chord of circle of R radius.

$$\frac{l_2}{2R} = \frac{a_1}{l_2} = \cos\alpha \qquad \text{(1)}$$

$$R = \frac{l_2^2}{2a_1} = \frac{l_1^2 + a_1^2}{2a_1} \qquad \text{(2)}$$

Rearranging and multiplying by a results in:

$$a/R = \frac{D_1 a_1}{l_1^2 + a_1^2} \qquad \text{(3)}$$

A NONLINEAR DIFFERENTIAL EQUATION FOR THE EVALUATION OF SOLID DAMPING

By G. M. Smith[1] and D. Albee[2]

Synopsis

This paper describes a nonlinear differential equation for the evaluation of solid damping of nonviscoelastic materials. Rectangular and circular cantilever beams are reduced to equivalent spring and mass systems in which the solid damping is considered to be proportional to the product of displacement and velocity. The free damped vibrations of beams of various lengths are recorded on oscillograph tapes. Damping parameters are then evaluated by matching oscillograph tapes of analog computer solutions with corresponding oscillograph tapes of the free damped vibrations of the test specimens. The results presented in this paper indicate that the ratio of the damping coefficient, α, to equivalent mass, m_e, is affected by the cross-sectional shape and natural frequency.

Solid Damping

Solid damping is the phenomenon whereby elastic and inelastic materials dissipate energy when subjected to vibrations, either free or forced. In highly elastic materials, such as steel, the energy dissipated by damping is quite small, while in inelastic materials, such as lead, this energy dissipation is quite large. Such energy is dissipated within the material itself by some internal mechanism.

The mechanism of solid damping is not well understood, and as a result various mechanical models have been devised to simulate the mechanism and behavior of materials as associated with deformation and time (**1**).[3] The basic units for the formulation of mechanical models are the Maxwell unit and the Kelvin unit, shown in Fig. 1. In each unit, the perfectly elastic spring simulates elastic deformation, and the dashpot containing a viscous fluid simulates viscous deformation. Various mechanical models may be formulated by various combinations of the basic units, but mechanical models made up of linear dissipative elements cannot be expected to reproduce closely the action of materials whose behavior is nonlinear. The nonlinear nature of materials may be approximated more closely by the introduction of nonlinear restoring and dissipative mechanisms into the basic units. The principal difficulty in the use of nonlinear models is that of obtaining solutions to the nonlinear differential equations.

For a visco-elastic material that behaves according to the Kelvin unit, the stress, σ, at a given instant depends not only on the magnitude of the strain, ϵ, but also on the strain rate, $\dot{\epsilon}$. For such a material the stress may be expressed mathematically as

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[1] Professor of Engineering Mechanics, University of Nebraska, Lincoln, Nebr.

[2] Martin Aircraft Corp., Denver, Colo.

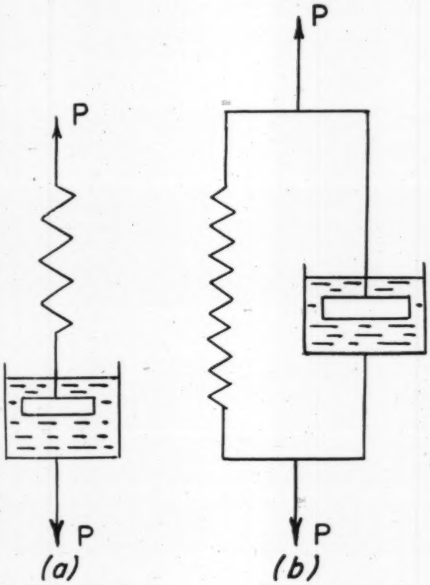
[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

$$\sigma = E\epsilon + F\dot{\epsilon} \ldots\ldots\ldots\ldots (1)$$

where:

E = modulus of elasticity, and

F = a constant related to a specific material.



(*a*) Maxwell unit (*b*) Kelvin unit

FIG. 1.—Basic Units for Mechanical Models.

Combining Eq 1 with beam theory, the differential equation for the free damped vibrations of a beam is found to be (2):

$$EI\frac{\partial^4 y}{\partial x^4} + FI\frac{\partial^5 y}{\partial x^4\,\partial t} + \gamma\frac{\partial^2 y}{\partial t^2} = 0 \ldots (2)$$

The second term of Eq 2 is the damping term. Solving Eq 2 by the method of separating variables, we find the damping parameter, n, in terms of the circular frequency, p, to be:

$$n = \frac{p^2 F}{2E} \ldots\ldots\ldots\ldots (3)$$

Unfortunately most structural materials do not follow the simple relation of Eq 3. Therefore, the damping characteristics of materials have usually been evaluated on the basis of an equivalent damping coefficient, n_e, as described in the linear differential equation for a simple spring and mass system:

$$\left.\begin{aligned} m\ddot{y} + c\dot{y} + ky &= 0 \\ \ddot{y} + 2n_e\dot{y} + p^2 y &= 0 \end{aligned}\right\} \ldots\ldots (4)$$

where:

y = linear displacement,

m = mass,

c = viscous damping constant,

$n_e = c/m$ = equivalent viscous damping coefficient,

k = spring constant, and

$p^2 = k/m$ = square of the circular frequency.

For free damped vibrations n_e is evaluated by measuring the rate of decay of the vibrations (3). The rate of decay is indicated by:

$$\frac{y_i}{y_{i+s}} = e^{n_e s\tau} \ldots\ldots\ldots\ldots (5)$$

where:

y_i = amplitude of vibration of the ith cycle,

s = number of cycles after the ith cycle,

y_{i+s} = amplitude of the $i + s$ cycle, and

τ = period of vibration.

In the case of forced vibrations with small damping, the apparent value of n_e may be determined from the simple relation:

$$n_e = \frac{p}{2\,\text{M.F.}} \ldots\ldots\ldots\ldots (6)$$

where:

M.F. = magnification factor at resonance.

Although it is generally recognized that the solid damping of most structural materials is not viscoelastic in nature, the expressions of Eqs 3, 5, and 6 are frequently used to evaluate the data obtained from cantilever beam specimens. Such a procedure for nonviscoelastic materials will yield results which are in essence only qualitative, since n_e has been found to vary in an obscure manner with the amplitude and frequency. Therefore, considerable difficulty has been encountered in the evaluation and comparison of materials as to their damping properties.

NONLINEAR MATHEMATICAL MODEL

The following equation may be used to evaluate the damping properties of nonviscoelastic materials:

$$m\ddot{y} + \alpha \mid y \mid \dot{y} + ky = 0 \ldots\ldots (7)$$

where α is a damping coefficient that depends upon the shape of the cross-section and length of the test specimen. The absolute value of the displacement, y, must be used in order to preserve the sign of $\dot{y}$, since the quantity $\alpha \left|y\right| \dot{y}$ must have the sign of $\dot{y}$ in order to remove energy from the system. Equation 7 may be interpreted as viscous damping with the variable coefficient $\alpha \left|y\right|$. It is noted that Eq 7 indicates that the damping is a function of the amplitude, that is, as the amplitude decreases the rate of decay also decreases. This is a typical characteristic of nonviscoelastic materials.

The application of Eq 7 to a cantilever beam, shown in Fig. 2, is accomplished by reducing the system to an equivalent spring and mass system as shown. The equivalent mass, m_e, considered concentrated at the end of the cantilever, is determined from Rayleigh's method (3). Taking the dynamic deflection as that of a statically loaded beam at the end, the equivalent mass, m_e, and spring constant, k, are found to be:

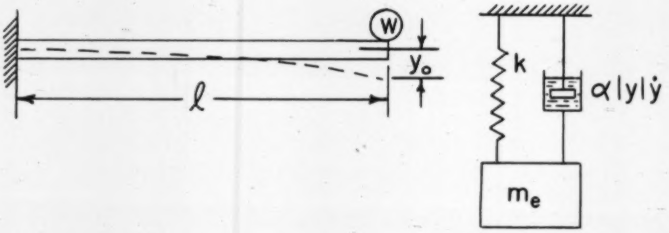


FIG. 2.—Cantilever Beam and Equivalent System.

$$m_e = \frac{1}{4}\left(\frac{33}{35} m_b + \frac{4W}{g}\right) \ldots\ldots (8)$$

$$k = \frac{3EI}{l^3} \ldots\ldots\ldots\ldots (9)$$

where:

m_b = mass of the beam, and
W/g = concentrated mass attached at end of beam.

In terms of the equivalent system, the nonlinear differential equation is written in the form:

$$\ddot{y} + \frac{\alpha}{m_e} \mid y \mid \dot{y} + \frac{k}{m_e} y = 0$$

or:

$$\ddot{y} + \frac{\alpha}{m_e} \mid y \mid \dot{y} + p^2 y = 0 \ldots (10)$$

TEST PROCEDURE

The data were obtained from the free damped vibrations of cantilever beams of 6061-T4 aluminum. The cross-sections of the specimens were a 1-in. diameter circle, a 0.748 by 0.875-in. rectangle, and a 0.49 by 1.29-in. rectangle. To obtain a range of frequencies for each cross-section, the weight, *W*, and length, *l*, (Fig. 2) were varied in each case. To ensure complete fixation at the fixed end so as to prevent a transfer of energy to the support, the specimens were placed in split clamping blocks which were subjected to a 40,000-lb load by a hydraulic testing machine.

Each specimen was excited at its fundamental natural frequency by suspending a weight from the free end by a string which was then cut, allowing the beam to vibrate. The initial amplitude at the free end, due to the suspended weight, was noted in each case for programming to the analog computer. In order to evaluate the effect of initial amplitude or stress level on the damping parameter, α/m_e, the initial amplitude was varied by changing the magnitude of the suspended weight.

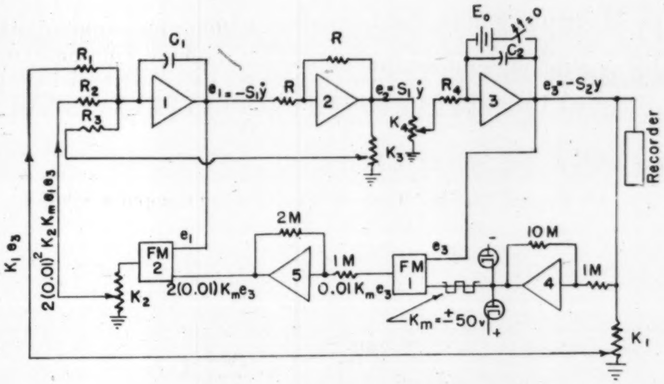


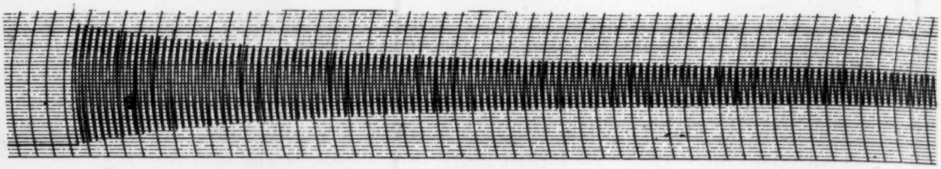
FIG. 3.—Analog Computer Circuit.

The output of SR-4 electric strain gages on the specimen was fed into an oscillograph for recording the free damped vibrations on oscillograph tapes. It was found that the extraneous damping of the small lead wires was negligible when the wires were glued to the surface of the specimen.

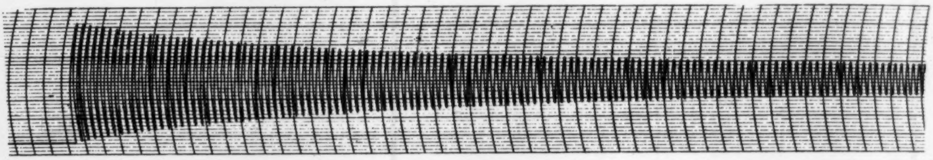
The natural period of vibration was measured to the nearest 0.0001 sec by an electronic counter which was triggered by the output of the strain gages.

ANALOG COMPUTER COMPUTATION

The evaluation of α/m_e for a given specimen and frequency was accomplished by programming Eq 10 to an analog computer. The computer circuit is shown in Fig. 3. Although the circuit is formulated from the fundamental principles of analog computation (3), certain points should be emphasized. The initial voltage, E_0, across capacitor C_2 is equal to the scale factor, S_2, times the initial deflection, y_0, of the free end of the cantilever beam. The damping term, $\alpha/m_e \mid y \mid \dot{y}$, is generated by the network of amplifiers *4* and *5* and the function multipliers *1* and *2*. In order to compensate for the small amount of internal circuit damping of the computer, the proper amount of negative damping is fed back into the system by adjusting the potentiometer K_3 to that of a conservative system ($K_2 = 0$ for a conservative system). The restoring force, *ky*, is simulated by the feedback through po-



(*a*) Free damped vibrations of specimen (1.29 by 0.49 in.) where:
$W = 0.625$ lb, $\tau = 0.0774$ sec, and
$y_0 = 1.15$ in., $p = 81.14$ radians per sec.
$l = 27$ in.,



(*b*) Computer solution: $\alpha/m_e = 1.12$ (in. sec)$^{-1}$.

FIG. 4.—Typical Matched Tapes of Specimen and Computer Solution.

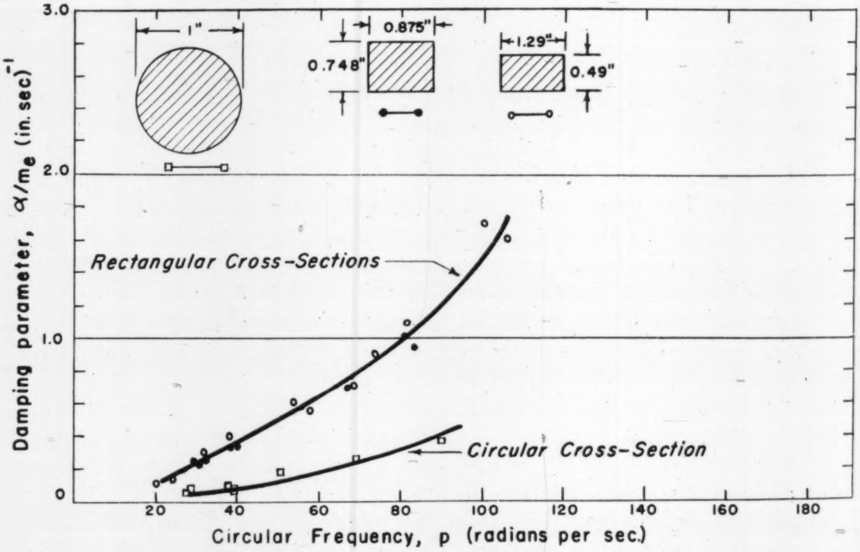


FIG. 5.—Variation of Damping Parameter with Circular Frequency.

tentiometer K_1. The relations between the physical quantities of Eq 10 and the computer circuit are as follows:

$$p = K_1/R_1C_1 = K_4/R_4C_2 \ldots\ldots (11)$$

$$E_0 = S_2y_0 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (12)$$

$$\frac{\alpha}{m_e} = \frac{S_2K_2}{R_2C_1}(2)(0.01)^2(50) \ldots\ldots\ldots (13)$$

The values of K_1, R_1, and C_1 are so selected that the frequency and period agree precisely with that of the specimen being programmed. The amplitude of the initial cycle from the computer solution is matched with that of the initial cycle of the specimen by the scale factor, S_2, and amplifier gain for the oscillograph.

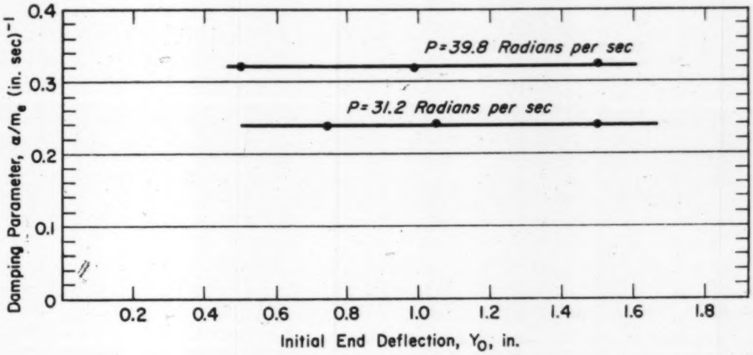


FIG. 6.—Variation of Damping Parameter with Initial End Deflection.

The value of α/m_e is then determined by a trial-and-error adjustment of the potentiometer setting, K_2, until the oscillograph tape from the computer is identical to that of the specimen. Typical matching tapes are shown in Fig. 4. An examination of the tapes will show that the rate of decay is not constant but decreases as the amplitudes diminish. The results of all the tests, indicated in Fig. 5, show the variation of α/m_e with the circular frequency, p.

As previously mentioned, the initial amplitude, y_0, was varied for individual specimens. In all cases the value of α/m_e was found to be independent of the amplitude, y_0, as shown by the typical examples in Fig. 6.

SUMMARY

The evaluation of the damping properties of nonviscoelastic materials by the nonlinear differential equation, Eq 10, offers a new approach for the evaluation and comparison of the damping properties of materials. Although the method requires the use of an analog computer, it is believed that it gives results that are more quantitative than methods based upon the classical viscous damping theory.

The data shown in Figs. 5 and 6 indicate the following:

1. The parameter α/m_e is independent of amplitude and is, therefore, a constant for a given specimen and frequency.

2. For a given shape of cross-section, the effect of the natural frequency on the parameter α/m_e may be established. This is an important factor that should be considered when comparing the damping characteristics of different materials.

3. The shape of the cross-section may have an appreciable effect on the damping characteristics. It is apparent from

Fig. 5 that a rectangular cross-section is more effective in damping than a circular cross-section.

The nonlinear procedure presented here may be extended to forced vibrations by introducing a sinusoidal forcing function in the right-hand side of Eq 10. The forcing function would be simulated in the computer by a sinusoidal input to amplifier *1* (Fig. 3).

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SYMPOSIUM ON IMPURITIES IN STEAM

INTRODUCTION

By J. K. Rice[1]

The measurement of the impurities present in steam has progressed from methods employing a throttling calorimeter to methods employing a flame photometer. The sensitivity of the methods for impurities has increased from thousands of parts per billion to tenths of parts per billion. The steadily increasing pressures and temperatures at which steam power systems operate have dictated continuous improvement in these methods of measurement. The potential to carry-over depositing substances from boiler water has greatly increased while the tolerance of turbines to deposits has decreased to the vanishing point. Direct volatilization of salts into steam is now an important source of such depositing substances. It has become essential to know not only the volatility of the various salts encountered but also whether simple stoichiometry prevails for each salt. It is known for example that volatilization of silicate from boiler water into steam does not bring with it stoichiometric quantities of sodium. Is this true for any other salts? This is the problem to which Mr. Rubright addresses himself in his paper.

Much of the published data on the volatilities of the several common salts suffers from either an insufficient description of the technique employed, or a great variance with other published data, or both. An attempt to verify the volatility of at least one of the salts by performing tests in full-size boilers instead of in laboratory test vessels was the object of the research conducted by Messrs. Ulmer and Klein.

The sensitivity of the flame photometric method for measuring impurities in steam has literally opened Pandora's box by revealing the complete inadequacy of our steam sampling methods. It should have been obvious long ago that if salts in superheated steam deposited in turbines they should also deposit in sampling lines. It remained, however, for Messrs. Coulter and Cobb to diagnose the difficulty and to find a cure.

It is apparent that we are beginning to understand the mechanism of volatile transport of solids into saturated steam. It is also apparent that we are a long way from an understanding of the physical chemistry of electrolytes in solution in high-temperature steam. We seem to have no significant understanding of the dynamics of deposition from steam. If trace elements can alter crystal structures, if trace elements can cause supersaturation, why cannot trace substances in steam hinder deposition? There is obviously a great deal of work to be done.

The authors of the papers present new ideas, new data, add new techniques. It is hoped that their efforts will stimulate further research on impurities in steam.

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[1] Symposium Chairman; Consulting Engineer and President, Cyrus Wm. Rice & Co., Pittsburgh, Pa.

THE STOICHIOMETRY OF THE VAPOROUS CARRY-OVER OF SODIUM CHLORIDE FROM HIGH-PRESSURE BOILER WATER

BY M. M. RUBRIGHT[1]

SYNOPSIS

The phenomenon of vaporous carry-over of boiler water salts into steam has been investigated in the past for the magnitude of carry-over of selected inorganic salts, but these investigations have not experimentally demonstrated whether the salt carry-over is stoichiometric with respect to the cation and anion involved.

An investigation into this facet of the vaporous carry-over of sodium chloride from a dilute solution at 2500 psig was conducted, using equipment which had previously been proven to contribute no significant mechanical carry-over of boiler water salts into the steam. The results indicate that the vaporous carry-over of sodium chloride at 2500 psig is stoichiometric in nature. Sodium chloride at this pressure carries over either as a molecule, or as ions, with no preferential increase of either the sodium or chloride ions over the stoichiometric ratio.

Some preliminary work was performed on the mechanism of the vaporous carry-over of sodium chloride at 3000 psig. No definite conclusions could be made from the limited amount of data obtained.

Although most investigators have assumed that solids in boiler water vaporize into the steam as compounds, actual proof of this postulation has not been established. Some investigators theorize that, like silica, all boiler water salts carry over as ions or elemental oxides. Knowledge of the exact nature of this carry-over is of importance in interpreting analytical data of steam condensates and would contribute to a better understanding of the behavior of impurities in steam.

In November 1959, ASTM Committee D-19 on Industrial Water was allocated a grant from the ASTM Research Fund to investigate the mechanism of the vaporous carry-over of boiler water salts. The Research Center of The Babcock & Wilcox Co. submitted a proposal to perform this investigation using test equipment which had previously been proven useful in the study of the carry-over of silica from boiler water, and the proposal was accepted.

The investigation was to consist of two successive phases. The first phase entailed studying the vaporous carry-over of sodium chloride at 2500 psig, since this compound carries over in sufficient quantity to permit the use of proven conventional analytical procedures for both the sodium and chloride contents of the resulting condensate. The second phase, to be conducted if funds permitted, was to investigate the vaporous carry-over of sodium sulfate and sodium phosphate from boiler water at

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2500 psig, using flame photometric techniques to measure sodium in the condensates, and radioisotope tracers to measure the sulfates and phosphates, since conventional analytical procedures could not yield accurate results at the level expected in the condensates.

To our knowledge, this is the first reported investigation of vaporous carry-over in which the concentrations of both the cation and anion of the vaporized salt are determined. Some previous investigations involved the use of silica (**1–5**).[2] The boiler water used in some of these investigations was of a high pH, but the sodium level in the boiler water or condensates was not determined. Other investigators (**6**) have measured the concentration of the anion present in the condensate by means of radioisotopes and, assuming that the relationship between the cations and anions was stoichiometric, have calculated the carry-over. Straub and Grabowski (**2**) performed some investigative work on the carry-over of sodium chloride at 600 F by measuring the conductivity of the resulting condensate. In all cases, however, the magnitude of the carry-over was studied, not its mechanism.

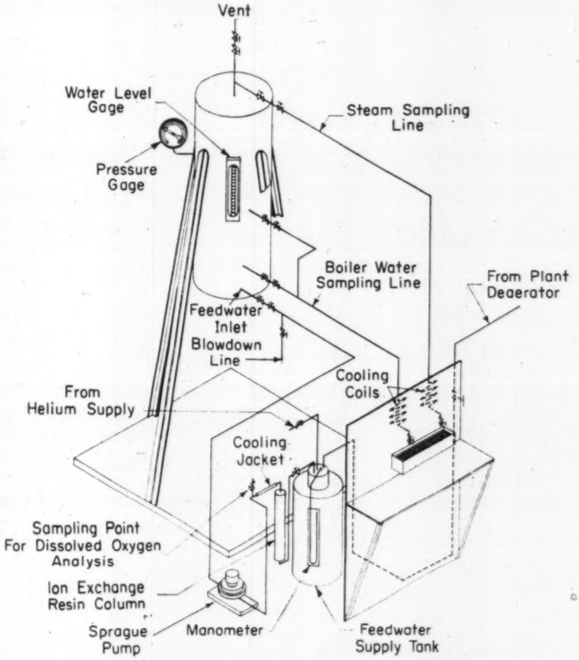
Vent
Water Level Gage
Steam Sampling Line
Pressure Gage
Boiler Water Sampling Line
From Plant Deaerator
Feedwater Inlet
Blowdown Line
Cooling Coils
From Helium Supply
Cooling Jacket
Sampling Point For Dissolved Oxygen Analysis
Ion Exchange Resin Column
Sprague Pump
Manometer
Feedwater Supply Tank

FIG. 1.—Test Equipment Arrangement.

The first part of the proposed investigation—that of studying the vaporous carry-over of sodium chloride at 2500 psig—was completed, along with some preliminary investigations of the vaporous carry-over at 3000 psig, in November 1960. The second phase of the investigation, involving a study of the carry-over of sodium sulfate and sodium phosphate at 2500 psig, was not started because of lack of funds.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

Details of the completed investigation are described in this report.

DESCRIPTION OF THE EQUIPMENT

The test equipment used consists of a small test boiler and accompanying sampling and feedwater supply systems. Figure 1 shows the general arrangement of the test equipment.

The design of the test boiler is shown in Fig. 2. This test boiler had previously been used in investigations of the vaporous carry-over of silica. During previous investigations, it had been proved that no significant mechanical carry-over of boiler water salts into the steam at 2500 psig occurs if the boiler is operated at steaming rates of 5 lb per hr or less (3). The boiler was designed for a maximum pressure of 3200 psig and a maximum steaming capacity of 50 lb per hr at 2500 psig. It is heated by electrical resistance heaters evenly spaced on the outside boiler surface below the water line. These heaters provide a maximum heat flux of 20,000 Btu per hr per sq ft. Power to the heaters is manually controlled.

A special feature of the test boiler is a concentric stainless steel liner, or shroud, extending from below the water line to the steam sampling point at the top. This liner is designed to prevent contact between surface condensate formed on the pressure vessel walls and the steam sample. Condensation occurring on the upper portion of the pressure vessel wall drains back to the boiler water in the annulus between the steam space liner and the pressure vessel walls. The liner thus provides an insulating space between the pressure vessel wall and the generated steam and minimizes the redissolving of vaporous boiler water salts in the steam space before sampling. The shroud skirt has four ½-in. holes evenly spaced around its circumference to improve circulation between the water in the annulus and the main body of water in the boiler.

The sampling system is arranged so that samples of both boiler water and

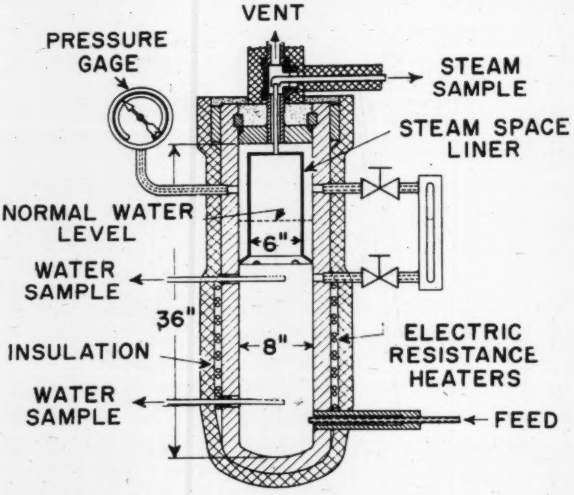

FIG. 2.—Test Boiler.

steam can be taken. Samples of the boiler water may be obtained at two different locations in the boiler—one at the bottom just above the feedwater inlet and the other at a point just below the shroud. The arrangement can be seen in Fig. 1. The sample lines are made of $\frac{1}{4}$-in. stainless steel and include cooling coils.

The feedwater supply is also shown in Fig. 1. Deaerated demineralized water is drawn from a plant supply line into a 13-gal polyethylene bottle. A positive pressure of helium is maintained above the water surface in the bottle at all times to minimize oxygen in-leakage.

A $\frac{1}{4}$-in. stainless steel line connects the feedwater bottle to the intake of a diaphragm pump. A column filled with a sulfited anion exchange resin and a water-cooled horizontal section are inserted in the line between the feedwater bottle and the pump. The water is pumped from the bottle, upward through the resin column (to remove traces of oxygen that might have been absorbed in the polyethylene bottle), passes through the water-cooled section (used for sample cooling during dissolved oxygen analysis) into the pump, and then is pumped into the boiler. A thermal sleeve in the boiler feedwater connection prevents thermal shock to the boiler from the cool feedwater.

A tee and valve are inserted in the feedwater line between the water-cooled section and the pump. Samples of the feedwater can be obtained at this point for dissolved oxygen analysis, and the sodium chloride solution used in the tests can be added here.

Another tee and valve are inserted in the feedwater line between the pump and the entry to the test boiler. A section of $\frac{1}{4}$-in. stainless steel tubing attached to the tee serves as a blowdown line and, after suitable cleaning, as a sampling point to obtain feedwater samples for analysis.

The feedwater used in these tests was sampled periodically and consistently assayed as containing less than 0.001 ppm Na, 0.1 ppm Cl, 0.001 ppm SiO_2, and 0.010 ppm dissolved oxygen.

TEST PROCEDURE

After several preliminary tests, the following procedure was followed in each test reported.

In the morning, the boiler water remaining from the previous day's test was removed from the boiler by a blowdown at approximately 400 psig pressure. This procedure was found necessary to prevent silica build-up in the boiler water from long-time operation at high pressures. The boiler was flushed with water for ten minutes and then drained again. The residual pressure in the boiler was relieved until it was less than 50 psig, and the boiler was then ready for addition of the new test solution.

The boiler was then filled to the proper water level with a sodium chloride solution (approximately 5000 ppm sodium chloride), prepared by metering concentrated sodium chloride solution into the deaerated demineralized feedwater as it was pumped into the boiler. Originally it was planned to use a concentration of 250 ppm of sodium chloride for these tests. During preliminary tests, however, it was learned that this concentration yielded condensates which did not contain enough sodium chloride to obtain reproducible analytical results. The concentration finally used on all the tests was 5000 ppm of sodium chloride.

The concentrated sodium chloride solution was prepared under essentially oxygen-free conditions in the following manner: A flask similar to those used in the dissolved oxygen procedure (ASTM Methods D 888 – 49 T)[3] was attached to

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[3] Methods of Test for Dissolved Oxygen in Industrial Water (Tentative) (D 888 – 49 T), 1958 Book of ASTM Standards, Part 10.

TABLE I.—TEST RESULTS FOR VAPOROUS CARRY-OVER AT 2500 PSIG.

	Test No. 1	Test No. 2	Test No. 3	Test No. 4	Test No. 5	Test No. 6	Test No. 7	Test No. 8	Test No. 9	Test No. 10
Boiler water at start of test[a]										
Na^+, ppm	1800	2000	1750	1750	1850	2000	2000	1700	1850	2150
Cl^-, ppm	2780	2940	2781	2870	2975	3075	2975	2600	2900	3375
SiO_2, ppm	7.5	8.5	6.0	6.0	6.0	6.5	5.5	5.5	5.5	6.0
NH_3, ppm	0	0.001	0	0	0	0	0	0.005	0	0
pH	8.1	8.2	7.41	7.42	7.3	7.2	7.71	7.72	8.52	7.8
Conductivity, mmhos/cm	8400	8880	8420	8620	8940	9300	8750	7850	8700	9770
Condensate 1										
Na^+, ppm	1.15	1.9	1.2	1.0	1.2	1.5	1.0	0.97	1.70	1.45
Cl^-, ppm	2.4	2.38	1.95	2.0	1.63	2.1	1.50	1.45	2.38	2.0
SiO_2, ppm	0.30	0.36	0.35	0.27	0.22	0.27	0.21	0.30	0.33	0.34
NH_3, ppm	0.30	0.005	0.001	0	0	0	0	0.001	0	0
pH	7.7	6.9	6.35	6.5	6.45	5.2	6.6	6.83	6.70	6.57
Conductivity, mmhos/cm	7.89	10.65	8.88	8.67	9.09	9.02	7.24	6.65	11.0	9.20
Condensate 2										
Na^+, ppm	1.15	1.0	0.7	1.2	1.55	0.9	1.3	1.20	1.40	1.45
Cl^-, ppm	2.2	1.63	1.10	2.0	2.3	1.36	2.2	1.88	2.08	2.12
SiO_2, ppm	0.48	0.40	0.33	0.30	0.29	0.28	0.37	0.35	0.36	0.38
NH_3, ppm	0.12	0.001	0	0	0	0	0	0.002	0	0
pH	7.0	6.45	7.6	6.45	6.6	6.25	7.2	6.65	6.52	6.55
Conductivity, mmhos/cm	8.06	6.45	5.96	8.93	11.0	7.42	9.98	7.91	8.91	9.86
Condensate 3										
Na^+, ppm	1.1	1.0	0.6	1.2	1.0	1.3	1.65	1.52	1.0	1.45
Cl^-, ppm	2.1	1.35	0.93	1.8	1.32	1.9	2.8	2.25	1.50	2.45
SiO_2, ppm	0.46	0.41	0.29	0.31	0.41	0.30	0.38	0.42	0.38	0.42
NH_3, ppm	0.08	0.001	0	0	0	0	0	0.004	0	0
pH	7.6	7.0	6.5	6.3	6.3	6.2	6.7	6.60	6.38	6.21
Conductivity, mmhos/cm	7.62	6.45	4.95	8.46	6.80	8.34	10.87	9.61	7.04	9.94

Condensate 4										
Na⁺, ppm	1.4	1.2	0.8	1.2	1.3	1.3	1.0	1.35	1.0	1.45
Cl⁻, ppm	2.4	1.65	1.17	1.9	1.9	1.93	1.63	2.0	1.38	2.32
SiO_2, ppm	0.46	0.46	0.30	0.40	0.41	0.33	0.35	0.46	0.38	0.44
NH_3, ppm	0.03	0	0	0	0	0	0	0	0	0
pH	7.6	6.8	6.48	6.43	6.3	6.1	7.8	6.52	6.30	6.1
Conductivity, mmhos/cm	9.07	7.91	6.88	8.79	7.95	9.09	7.58	8.87	7.26	9.68
Boiler water at end of test[a]										
Na⁺, ppm	2200	2400	2200	2200	2350	2500	2400	2150	2400	2800
Cl⁻, ppm	3540	3600	3400	3550	3680	3720	3575	3050	3650	4100
SiO_2, ppm	13.0	14.4	12.5	10.5	12.5	11.5	10.5	8.0	8.5	10.5
NH_3, ppm	0	0	0	0	0	0	0	0	0	0
pH	8.2	8.1	7.78	7.52	7.3	7.7	8.2	8.25	8.41	8.2
Conductivity, mmhos/cm	10 240	10 460	9970	10 510	10 620	10 840	10 440	9600	10 710	11 750
Boiler water at end of test[b]										
Na⁺, ppm	Sample not taken	2000	1850	2050	2050	2100	2200	1750	1800	2150
Cl⁻, ppm		3000	3019	3100	3200	3200	3150	2775	2775	3325
SiO_2, ppm		19.40	15.0	13.0	11.5	11.5	10.5	9.0	7.5	9.0
NH_3, ppm		0.003	0	0	0	0	0	0.005	0	0
pH		7.9	7.9	7.70	7.5	7.8	8.0	8.20	8.30	8.0
Conductivity, mmhos/cm		9000	9080	9510	9460	9570	9300	8430	8280	9750

Tests 1 through 7: Steaming Rate = 2 lb per hr, and Tests 8 through 10: Steaming Rate = 5 lb per hr.

[a] These samples were taken at a sampling location just below the shroud in the test boiler.

[b] These samples were taken at a sampling location near the bottom of the boiler.

the dissolved oxygen sampling tee in the feedwater line. The flask, previously filled with the required amount of crystalline sodium chloride, was mounted vertically, and the valve to the feedwater line opened. Feedwater was forced up into the flask by pressurizing the feedwater tank with helium. The flask could be completely filled with deaerated feedwater by adjustment of the sampling valve and the two stopcocks in the flask. The sodium chloride then easily dissolved in the water. In this manner, the concentrated sodium chloride solution was prepared without being exposed to air or oxygen-containing water.

The resulting sodium chloride solution thus formed was metered into the feedwater line in small amounts during the boiler-filling operation, yielding a homogeneous mixture of salt solution and feedwater, and avoiding segregation in the boiler during initial start-up operations.

In the 2500-psig tests, the test boiler was initially filled until the water level was at the midpoint of the gage-glass. Heat was then applied to the boiler. When the pressure in the boiler reached 1500 psig, a boiler water sample was withdrawn from the sampling location just below the shroud. The sample was analyzed for its pH and conductivity to insure that proper operating conditions existed before going to higher pressures.

When the boiler water conditions proved to be satisfactory, water was drained from the boiler until the water level was just at the top of the gage-glass. This was done to insure that the expansion of water occurring at high temperatures would not cause over-filling of the boiler, leading to contamination of the condensate sampling line.

The same procedure was followed in the 3000 psig tests, except that a measured quantity of water was placed in the test boiler at the start of the test, sufficient to permit the withdrawal of the necessary boiler water and steam samples without dropping the water level below the lower edge of the shroud during the test. This method was required because the water level gage was not connected during the 3000 psig tests.

When the pressure in the boiler reached the desired value, current to the electric heaters was regulated to maintain this pressure. After the operating pressure had been maintained for 1 hr, the steam sampling line was opened, adjusted to the proper steam flow, and the first liter of condensate collected and discarded. This was done to insure proper cleanliness in the condensate line. A sample of the boiler water was withdrawn from the sampling location just below the bottom of the shroud. Immediately afterward, four successive 250-ml samples of the steam condensate were collected. Another boiler water sample was withdrawn from the sampling location just below the shroud, and one was taken from the bottom of the test boiler. The steam sampling line was then closed, and heat to the boiler was reduced. The pressure in the boiler diminished overnight to approximately 400 psig, and the boiler was ready for the blowdown operation the following morning.

The boiler water and condensate samples were analyzed immediately for pH and conductivity. Analyses for the sodium, chloride, silica, and ammonia content were made the next day. The analytical procedures used in the investigation are listed in Appendix II.

TEST RESULTS

Table I lists the analytical results for the boiler water and condensate samples obtained on the various tests conducted at 2500 psig. In these tests, the nominal concentration of sodium chloride in the boiler water was 5000 ppm. In addition to sodium chloride, the boiler water contained silica (from 5.5 to 14.4 ppm), traces of sulfite and sulfate (resulting from leakage of sulfite from the resin column), and, in the early tests, traces of ammonia which had been left in the boiler from previous cleaning operations. The presence of sulfite in the boiler water was indicated by the appearance of one of its high-temperature decomposition products—hydrogen sulfide—in most of

TABLE II.—TEST RESULTS FOR VAPOROUS CARRY-OVER AT 3000 PSIG.

	Test No. 11	Test No. 12	Test No. 13	Test No. 14
Boiler water at start of test[a]				
Na^+, ppm	1710	1850	2050	1950
Cl^-, ppm	2750	2950	3100	3000
SiO_2, ppm	1.1	0.8	0.8	1.08
pH	6.9	7.0	7.8	6.9
Conductivity, mmhos/cm	8020	8210	9040	8880
Condensate 1				
Na^+, ppm	26.5	28.0	24	20.0
Cl^-, ppm	35.6	39.4	44.4	31.4
SiO_2, ppm	0.17	0.16	0.12	0.19
pH	5.9	5.85	6.0	5.8
Conductivity, mmhos/cm	123	134	152	116
Condensate 2				
Na^+, ppm	32.0	34.5	29	27.5
Cl^-, ppm	44.8	49.4	54.6	44.2
SiO_2, ppm	0.20	0.19	0.17	0.235
pH	5.85	5.85	5.9	5.7
Conductivity, mmhos/cm	153	167	185	157
Condensate 3				
Na^+, ppm	38.0	44.0	28.5	Sample not taken
Cl^-, ppm	54.8	61.8	53.2	
SiO_2, ppm	0.28	0.21	0.20	
pH	5.8	5.80	6.0	
Conductivity, mmhos/cm	185	210	180	
Condensate 4				
Na^+, ppm	33.0	40.0	23.5	Sample not taken
Cl^-, ppm	47.4	56.8	43.0	
SiO_2, ppm	0.30	0.21	0.21	
pH	5.75	5.80	5.9	
Conductivity, mmhos/cm	160	193	146	
Boiler water at end of test[a]				
Na^+, ppm	2500	2250	2950	2550
Cl^-, ppm	3900	3925	4425	3925
SiO_2, ppm	1.8	1.4	1.44	1.46
pH	7.4	7.1	8.4	6.7
Conductivity, mmhos/cm	10 510	10 740	11 950	10 960
Boiler water at end of test[b]				
Na^+, ppm	1800	1900	2150	1950
Cl^-, ppm	2850	2950	3325	3050
SiO_2, ppm	4.8	4.2	3.68	3.15
pH	7.3	8.0	8.5	8.1
Conductivity, mmhos/cm	8160	8840	9240	8900

Tests 11 through 14: Steaming Rate = 5 lb per hr.

[a] These samples were taken at a sampling location just below the shroud in the test boiler.

[b] These samples were taken at a sampling location near the bottom of the boiler.

TABLE III.—STOICHIOMETRY OF SODIUM CHLORIDE RATIOS AT 2500 PSIG.

Test No.	Cl^- Found, ppm	NH_3 Found, ppm	Corrected Cl^-, ppm	Theoretical Na^+ from Cl^- Value, ppm	Na^+ Found, ppm	Stoichiometric Ratio?[a]
No. 1						
Condensate 1	2.4	0.30	1.77	1.15	1.15	Yes
2	2.2	0.12	1.95	1.26	1.15	Yes
3	2.1	0.08	1.93	1.25	1.1	Yes
4	2.4	0.03	2.34	1.52	1.4	Yes
No. 2						
Condensate 1	2.38	0.005	2.38	1.54	1.9	No
2	1.63	0.001	1.63	1.06	1.0	Yes
3	1.35	0.001	1.35	0.87	1.0	Yes
4	1.65	0	1.65	1.06	1.2	Yes
No. 3						
Condensate 1	1.95	0	1.95	1.26	1.2	Yes
2	1.10	0	1.10	0.7	0.7	Yes
3	0.93	0	0.93	0.6	0.6	Yes
4	1.17	0	1.17	0.76	0.8	Yes
No. 4						
Condensate 1	2.0	0	2.0	1.30	1.0	No
2	2.0	0	2.0	1.30	1.2	Yes
3	1.8	0	1.8	1.17	1.2	Yes
4	1.9	0	1.9	1.10	1.2	Yes
No. 5						
Condensate 1	1.63	0	1.63	1.06	1.2	Yes
2	2.3	0	2.3	1.5	1.55	Yes
3	1.32	0	1.32	0.85	1.0	Yes
4	1.9	0	1.9	1.2	1.3	Yes
No. 6						
Condensate 1	2.1	0	2.1	1.36	1.5	Yes
2	1.36	0	1.36	0.88	0.9	Yes
3	1.9	0	1.9	1.2	1.3	Yes
4	1.93	0	1.93	1.25	1.3	Yes
No. 7						
Condensate 1	1.50	0	1.50	0.97	1.0	Yes
2	2.20	0	2.20	1.4	1.3	Yes
3	2.8	0	2.8	1.81	1.65	?
4	1.63	0	1.63	1.06	1.0	Yes
No. 8						
Condensate 1	1.45	0.005	1.45	0.94	0.97	Yes
2	1.88	0.001	1.88	1.22	1.20	Yes
3	2.25	0.002	2.24	1.45	1.52	Yes
4	2.0	0.004	2.0	1.30	1.35	Yes
No. 9						
Condensate 1	2.38	0	2.38	1.54	1.70	?
2	2.08	0	2.08	1.35	1.40	Yes
3	1.50	0	1.50	0.97	1.0	Yes
4	1.38	0	1.38	0.89	1.0	Yes
No. 10						
Condensate 1	2.0	0	2.0	1.30	1.45	?
2	2.12	0	2.12	1.37	1.45	Yes
3	2.45	0	2.45	1.58	1.45	Yes
4	2.32	0	2.32	1.50	1.45	Yes

[a] Limit of analytical error for Na^+ = 0.15 ppm.

the condensates. The maximum concentration of sulfite in the boiler water did not exceed 2 ppm, the lower limit of detection of the analytical method used as indicated by repeated analyses. Two steam flow rates were used—2 and 5 lb per hr.

Table II lists the results obtained on the tests conducted at 3000 psig. The boiler water used in these tests had the same nominal concentration of sodium chloride—5000 ppm—as that used in the 2500 psig tests. The boiler water, however, contained no ammonia and had a lower silica content because the gageglass was disconnected during these tests. A steam flow rate of 5 lb per hr was used in this tests.

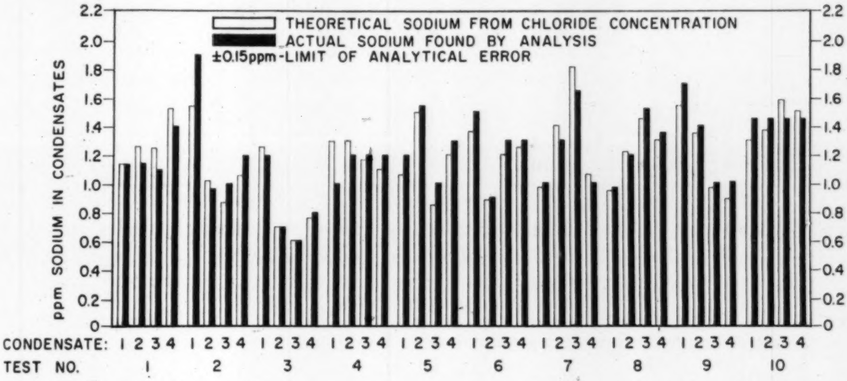

FIG. 3.—Comparison of Theoretical Versus Actual Sodium Content of Condensates—2500 psig Tests

DISCUSSION OF RESULTS

Tests Conducted at 2500 psig:

Table III lists the sodium and chloride contents of the steam condensates obtained from the tests conducted at 2500 psig. Included in this table is the quantity of sodium which would theoretically combine stoichiometrically with that amount of chloride found in the condensate. Figure 3 is a graphical comparison of the theoretical and actual sodium values obtained. In test No. 1, in which relatively large amounts of ammonia were present in the condensate samples, the chloride values were corrected to account for the chloride which could theoretically be present as ammonium chloride. The limit of error in the sodium analyses is assumed to be ±0.1 to 0.15 ppm.

The ratio of sodium to chloride found in the condensates agrees well with the stoichiometric ratio of sodium chloride. This relationship holds well even in the condensates from test No. 1 after the chloride values are corrected for the ammonia content of the condensates. It appears from these results that sodium chloride vaporizes from boiler water at 2500 psig either as molecules or as ions in stoichiometric ratios.

No indications were found that sodium chloride volatilizes in any form other than as sodium chloride molecules or as equal numbers of sodium and chloride ions.

Theoretically, the magnitude of vaporous carry-over is determined by comparing the concentration of sodium chloride in the condensate with the concentration of sodium chloride in its corresponding boiler water. If this procedure is followed with the experimental results obtained in this series of tests, however, the magnitude of carry-over found (for either sodium chloride or silica) is lower than that found by previous investigators.

The reasons for these apparent discrepancies are many and are related to the very nature of this investigation. The prime purpose of this project was the study of the mechanism of vaporous carry-over. The test equipment was operated in such a manner that the condensate samples obtained would be meaningful for mechanism studies. The determination of the magnitude of carry-over was of secondary importance.

TABLE IV.—STOICHIOMETRY OF SODIUM CHLORIDE RATIOS AT 3000 PSIG.

Test No.	Cl^- Found, ppm	Theoretical Na^+ from Cl^- Values, ppm	Na^+ Found, ppm	Stoichiometric Ratio?[b]	Excess Na^+, ppm	Excess Na^+, per cent
No. 11						
Condensate 1	35.6	23.1	25.5	No	+2.4	+10.4
2	44.8	29.0	32.5	No	+3.5	+10.7
3	54.8	36.5	39.0	No	+2.5	+6.4
4	47.4	30.7	34.5	No	+3.8	+11.0
No. 12						
Condensate 1	39.4	25.5	28.0	No	+2.5	+8.9
2	49.4	32.0	34.5	No	+2.5	+7.2
3	61.8	40.0	44.0	No	+4.0	+9.1
4	56.8	36.8	40.0	No	+3.2	+8.0
No. 13						
Condensate 1	44.4	28.8	29.0	Yes	...	...
2	54.6	35.4	35.5	Yes	...	...
3	53.2	34.5	34.5	Yes	...	...
4	43.0	27.9	28.5	?	...	...
No. 14						
Condensate 1	31.4	20.3	20.0	Yes	...	...
2	44.2	28.6	27.5	No	−1.1	−3.8
PRELIMINARY TESTS						
Test A						
Condensate 1	64	41.4	53	No	+11.6	+28
2	88	57.0	64	No	+7.0	+12.3
3	120	78.8	84	No	+5.2	+6.6
4	136	87.1	92	No	+4.9	+5.6
5	133	86.2	91	No	+4.8	+5.6
6[a]	126	81.6	88	No	+6.4	+7.8
Test B						
Condensate 1	36	23.3	23.5	Yes	...	...
2	32	20.7	26	No	+5.3	+25.6
3	34	22.0	27.5	No	+5.5	+25
4	28	18.1	23.5	No	+5.4	+30
5[a]	28	18.1	25	No	+6.9	+38

[a] Because of the uncertainty of water levels at the start of these tests, these were actually condensate samples, although collected as boiler water samples.

[b] Limit of analytical error for $Na^+ = \pm 0.5$ ppm.

A complete discussion of the conditions required to obtain valid answers in the mechanism studies, and the effect that the establishment of such conditions has upon obtaining the magnitude of vaporous carry-over appears in Appendix I.

The over-all effect of these conditions is that the exact concentration of the boiler water corresponding to a given condensate sample cannot be determined, and an accurate calculation of the magnitude of vaporous carry-over cannot be made.

However, it does appear that the magnitude of sodium chloride vaporous carry-over at 2500 psig may be lower than should be expected. Perhaps this particular feature should be reinvestigated at some future date using techniques specifically designed for magnitude studies.

The actual form of the vaporous carry-over cannot definitely be ascertained from this investigation. Since the steam samples containing the sodium chloride necessarily had to be condensed before analysis, the exact form of the sodium chloride in the steam—whether it existed as sodium chloride molecules or as equal numbers of sodium and chloride ions—cannot be determined.

It does appear that such carry-over is not the result of the vapor pressure of solid sodium chloride. At 2500 psig (688 F), the vapor pressure of solid sodium chloride is in the order of 2×10^{-7} mm Hg, which corresponds to a condensate concentration of 4×10^{-7} ppm under these test conditions.

The fact that the ratio of sodium to chloride in the condensates is stoichiometric does give increased reliability to the calculation of the concentration of sodium chloride in condensates from the measurement of one of the constituents. This adds support to the use of techniques such as flame photometry for analyzing impurities in condensates, at least for sodium chloride.

At the same time, the tests also indicate the effect that relatively small amounts of impurities in the boiler water may have upon the nature of the solids in the condensate. A good example of this is the effect that small amounts of ammonia had on the vaporous carry-over of chlorides in test No. 1. These tests emphasize the importance of having accurate knowledge of the concentrations of all constituents in the boiler water before attempting to evaluate condensate analytical data.

Tests Conducted at 3000 psig:

Table IV lists the sodium and chloride contents of the condensates obtained in the tests conducted at 3000 psig. It is arranged similarly to Table III, including the theoretical amounts of sodium in the condensates calculated from the chloride contents of the condensates. Neither the boiler water nor the condensates contained any ammonia, and no corrections were applied to the chloride results.

The results of these tests, in contrast to those obtained for the 2500-psig tests, showed no clear-cut evidence that a stoichiometric ratio of sodium to chloride exists in the condensate, although the condensates for tests Nos. 13 and 14 did exhibit reasonable stoichiometric ratios.

The other condensates contained a large excess of sodium over that required for combination with the chloride. The reason for this discrepancy is not known. One explanation for this would be the combination of the excess sodium with some other anion. The pH of the condensates was low (5.7 to 5.9), and it was suspected that an anion of the strong acid type was present. The number of eligible anions is small—for example, bisulfates or bisulfites. The presence of sulfites and sulfates in the condensates was checked on several of the condensates. The maximum concentration found was less than 0.5 ppm, which would not account for the excess sodium, neglecting the excess sulfite or sulfate needed to lower the pH to the figures obtained.

It appears that the source of the non-stoichiometry was present in the first tests at 3000 psig and that the source gradually diminished during subsequent tests, although the pH of the condensates remained low, even for those which possessed approximately stoichiometric amounts of sodium.

The tests conducted at 3000 psig were preliminary, and no definite conclusions concerning the mechanism of vaporous carry-over should be drawn from the results of these tests. It would be desirable to perform additional tests at this pressure to determine whether the mechanism of carry-over is different than that occurring under 2500-psig conditions.

CONCLUSIONS AND RECOMMENDATIONS

1. The results of the tests conducted at 2500 psig indicate that the vaporous carry-over of sodium chloride at this pressure is stoichiometric and that the mechanism is molecular in nature, or, if ionic, is such that sodium and chloride ions carry-over in equal numbers.

2. The test conditions required for mechanism studies in the test equipment used preclude any accurate determination of the magnitude of sodium chloride vaporous carry-over.

3. In spite of the uncertainties involved in calculating the magnitude of vaporous carry-over, the general levels of total vaporous carry-over obtained in this test were much lower than those obtained in previous investigations. This particular phase of vaporous carry-over should be re-investigated at some future date, using techniques specifically designed for magnitude studies. Provisions should be made to insure homogeneity of the boiler water in these studies.

4. No definite conclusions can be made concerning the mechanism of vaporous carry-over of sodium chloride at 3000 psig because of the limited number of tests made.

5. Further work to determine the mechanism of vaporous carry-over of other sodium salts of strong acids such as sodium sulfate and sodium phosphate is recommended.

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APPENDIX I

DETERMINATION OF THE MAGNITUDE OF VAPOROUS CARRY-OVER

The greatest potential source of error in the study of the mechanism of vaporous carry-over is mechanical carry-over of boiler water salts into the steam samples. Mechanical carry-over, in addition to increasing the apparent magnitude of vaporous carry-over, also tends to mask any real deviations in the ratio of cations to anions in the condensate from the stoichiometric ratio. The prevention, or at least the minimizing, of mechanical carry-over can be obtained with the test boiler used in these tests by using a suitable steaming rate, maintaining a semi-stagnant boiler water surface, and by the presence of a large steam space above the water level in the test boiler. The establishment of some of these conditions, however, has a deleterious effect upon the accurate determination of the magnitude of vaporous carry-over.

It had previously been demonstrated (**3**) that no significant mechanical carry-over occurs if the steaming rate of the test boiler is 5 lb per hr or less. The validity of this operating principle has been reaffirmed in the present investigation. At the same time, another potential source of error was found not to be present.

The test boiler used in this investigation (shown in Fig. 2) contains a stainless steel liner. The annular space between the liner and the walls of the pressure vessel prevents contact between the steam sample and the surface condensate formed on the vessel walls and minimizes redissolving the vaporous boiler water salts in the steam space before sampling. The steam sampling line is not directly connected to the liner. Instead, the up-stream end of the steam sampling line consists of a downward-turned elbow, the diameter of which is larger than that of the steam line from the liner. The liner steam line projects up into this elbow but is not directly connected to the steam sampling line. This arrangement permits equalization of steam pressures between the liner and the annulus and assures equal boiler water levels in both regions. Since the larger part of the steam generated in the test boiler is produced inside the liner, with only a small fraction generated in the annulus, the direction of the steam flow between the two regions is toward the annulus. At low steam flows, however, the probability exists that some of the steam from the annulus might diffuse into the steam sampling line. The steam present in the annulus must be considered to be washed steam, since it has been in contact with water condensed on the vessel walls, and its presence would cause a decrease in the concentration of vaporous boiler water salts in the resulting steam sample. Back diffusion and the resulting dilution of the liner steam sample would decrease as the steaming rate increases.

If these conditions exist at the steaming rates tested—mechanical carry-over and back-diffusion of washed steam into the steam sampling line—the sodium chloride content of the condensate should increase as the steaming rate increases. Two different steaming rates were used in these tests—2 and 5 lb per hr. No significant differences were found in the condensates from the two steam flow rates. This indicates that the steam flows used were appropriate and reaffirmed the operating characteristics found by previous users of the test equipment.

The other two conditions maintained in this investigation to prevent mechanical carry-over—the presence of a large steam space above the water level in the test boiler and the presence of a semi-stagnant boiler water surface—have a deleterious effect upon the determination of the magnitude of vaporous carry-over.

The large steam space in the test boiler causes a distinct lag between the time a unit volume of steam vaporizes from the boiler water surface and the time it leaves the test boiler. The steam residence time in the test boiler may be as much as four times as long as the sample collection time at the steam flows used. In the 2500-psig tests, using a steam flow of 5 lb per hr, it took 6 min to collect a 250-ml condensate sample. It required, theoretically, a maximum time of from 22 to 26 min for passage of the corresponding volume of steam from the boiler water surface to the exit of the boiler with an additional 1 to 2 min for the sample to traverse the sampling line. Use of a different steam flow would alter the actual residence and sample collection times, but not the ratio. This means that a condensate sample actually leaves the boiler water surface as steam appreciably before the time it is collected as a sample, and condensates should be compared with conditions in the boiler water at the time of vaporization, which in the case of the 5 lb per hr steam flow may be as much as 22 to 26 min before the condensate is sampled.

This would be of little importance if the concentration of sodium chloride in the boiler water remained constant throughout the test. In the tests reported here, however, this is not true. The concentration of sodium chloride in the boiler water increases during the test because of the evaporation of the steam collected as the condensate sample.

In addition, the test boiler demonstrates limited thermal circulation between its upper and lower regons, as indicated by the large differences in sodium chloride content in boiler water samples obtained at the two different sampling locations, as shown in Table I. There is probably also a difference between the very top layer of the boiler water and the region from which the boiler water samples are taken (immediately below the shroud). This condition is caused by the more-or-less isolated volume of water in the shroud becoming concentrated by evaporation, with no corresponding influx of less concentrated boiler water from below. This makes it difficult to appraise accurately the concentration of sodium chloride in the water from which a specific condensate sample is obtained.

The possibility that there is mixing of the steam in the steam space during the test run is problematical. The long steam residence time should tend to promote this, although the pressures existing in this space should diminish it. The effect of such mixing would be to integrate the various steam samples and make it even more difficult to appraise the magnitude of carry-over. The more-or-less randomness of the sodium chloride content of the condensates obtained at 2500 psig (which do not tend to increase in carry-over content as the test progresses and the boiler water becomes more concentrated) indicates that this does happen, although such is not the case in the 3000 psig tests.

These two conditions—the long steam residence time (which yields apparently high boiler water concentrations) and the poor thermal circulation (which yields apparently low boiler water concentrations)—make it difficult to determine the exact concentration of sodium chloride in the boiler water corresponding to a given condensate sample, and any calculations of magnitude of vaporous carry-over from the test data must be made with caution.

APPENDIX II

ANALYTICAL METHODS

The following analytical procedures were used in this investigation:

1. *Sodium* was determined in both the boiler water and condensate samples with a Beckman flame photometer, using the 330 and 589 mμ lines, compared to standards prepared from sodium chloride (ASTM Methods D 1428 – 56 T).[4]

2. *Chlorides* were determined in the boiler water sample by the Mohr titration procedure, identical to that of ASTM Methods D 512 – 55 T.[5]

Chlorides were determined in the condensates from the 2500-psig tests by the mercuric thiocyanate ferric-alum procedure.

Chlorides were determined in the condensates from the 3000-psig tests by the mercuric nitrate titration procedure (ASTM Methods D 512 – 55 T).[5] This method was used because the condensates contained too much chloride to use the mercuric thiocyanate ferric-alum colorimetric procedure.

3. *Silica* was determined in both the boiler water and condensate samples by the silica-molybdate method (ASTM Methods D 859 – 55 T[6] and D 1689 – 59 T).[7]

4. *Ammonia* was determined in both the boiler water and condensate samples with Nessler's reagent (ASTM Methods D 1426 – 58).[8]

5. *Dissolved Oxygen* in the feedwater was determined according to ASTM Methods D 888 – 49 T.[3]

6. *pH* of the boiler water and condensate samples was determined with a Beckman Zeromatic pH meter according to ASTM Method E 70 – 52 T.[9]

7. *Conductivity* of the boiler water and condensate samples was determined with a Leeds and Northrup conductivity bridge according to ASTM Methods D 1125 – 59 T.[10]

Hydrogen sulfide (resulting from the decomposition of sulfite at high temperatures in the test boiler) was present in some of the condensates. Since sulfides interfere in both the mercuric thiocyanate colorimetric and mercuric nitrate titration procedures, the samples containing sulfides were treated with 30 per cent hydrogen peroxide and boiled for several minutes before analysis. This modification was checked with standards and found to contribute no noticeable errors in either of the procedures.

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[8] Methods of Test for Ammonia in Industrial Water and Industrial Waste Water (D 1426 – 58), 1958 Book of ASTM Standards, Part 10.

[9] Method of Determination of pH of Aqueous Solutions with the Glass Electrode (Tentative) (E 70 – 52 T), 1958 Book of ASTM Standards, Parts 6 through 10.

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THE PREVENTION OF ERRORS IN STEAM PURITY MEASUREMENT CAUSED BY DEPOSITION OF IMPURITIES IN SAMPLING LINES

By R. V. Cobb[1] and E. E. Coulter[1]

Synopsis

The measured impurity concentrations of superheated steam samples were less than one tenth of those in saturated steam samples from the same boiler. An investigation of this phenomenon revealed that a major portion of the impurities in the superheated steam samples deposited on the sampling line surfaces. This deposition is apparently caused by the decreasing ability of superheated steam to retain impurities in vaporous form as steam temperature is reduced. Since an uninsulated steam sampling line loses heat to its surroundings, the deposits form on this cooler surface. A sampling system was developed that eliminates such deposition and its attendant error.

With the gradual improvement of separating equipment, the steam produced by boilers for use in power generation is an extremely pure substance. Its impurity content, for example, may be only a fraction of a part per million. Nevertheless, a boiler that produces more than a million pounds of steam per hour may pass several hundred pounds of impurities to a turbine in a year's time. These impurities represent a potential turbine deposit that may lead to appreciable losses of capacity and efficiency. Consequently, the control of conditions affecting steam contamination becomes a significant factor in boiler operation and requires an accurate and reliable measure of the impurities in steam.

Within the past decade, new analytical methods and instrumentation for measuring impurities in steam have been developed, such as the sodium tracer technique (**1-3**)[2], that enable measurement in the parts-per-billion range. However, an analytical method having high sensitivity and accuracy is of little value if the sample analyzed is not representative of the material being sampled, and the representative sampling of steam is a very difficult task.

Steam sampling has been the subject of many research investigations, but most of these have been concerned with the problem of obtaining a representative sample of saturated steam. Impurities in saturated steam consist of minute droplets of boiler water of unknown spectrum and distribution and, at higher pressures, boiler-water constituents in vaporous form. Furthermore, many of the entrained droplets may impinge on the steam pipe, forming a liquid film that flows along the pipe surface. These conditions present a formidable problem in obtaining a representative sample.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

Many people associated with steam sampling problems feel that sampling superheated steam will eliminate these problems and that a truly representative sample is obtained, because no liquid film will exist on the surface of the pipe, and the impurities in superheated steam will occur as a vapor or as extremely fine dust. Although a representative sample is obtained, the authors' experiences show that sampling superheated steam by conventional methods leads to very large errors, for the impurities tend to deposit on the sampling line surfaces. These experiences are discussed, and sampling procedures and equipment necessary to avoid errors due to sampling line deposits are described.

THE PROBLEM

After the initial data on the application of the sodium tracer method for measuring impurities in steam were published (1), the instrumentation was further developed during 1956 to enable the continuous analysis and recording of the sodium content of a flowing sample. This improved apparatus has been used extensively, both in the laboratory and in the field, and the information obtained has been very useful in evaluating the performance of steam separating equipment and in emphasizing the extremely low amount of carry-over from modern boilers operating at normal conditions.

However, all of these early tests were concerned with carry-over in saturated steam samples. Early in 1957, an attempt to monitor the impurities concentration in steam throughout a boiler-turbine system resulted in unusually large differences between the sodium content of saturated and superheated steam. The steam sampling locations are shown schematically in Fig. 1. They include (1) a sample of saturated steam from the steam drum outlet, (2) a slightly superheated sample from the superheater inlet header, and samples of (3) superheated steam taken after the first-pass superheater, (4) after the second-pass superheater, (5) from the steam pipe before the plant main steam line, (6) from the turbine inlet, and from (7) the turbine outlet. The entire plant consisted of eight boilers and eight turbines operating at a

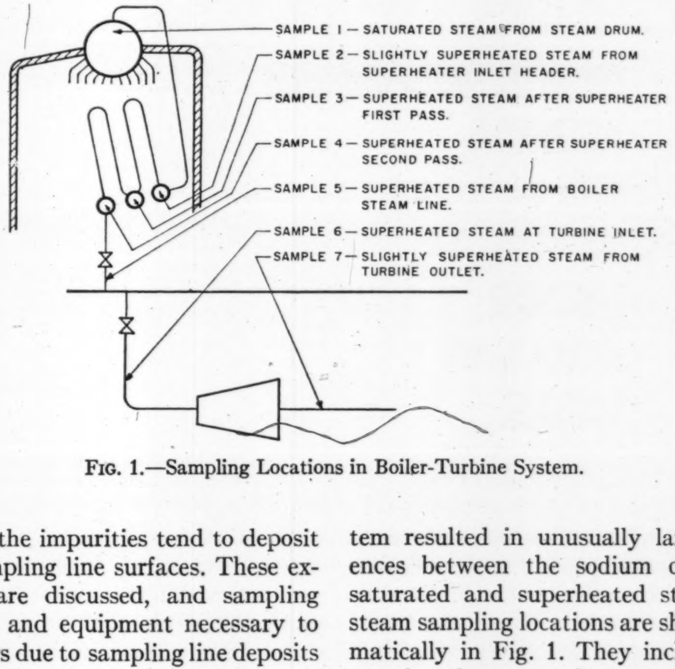
SAMPLE 1 — SATURATED STEAM FROM STEAM DRUM.
SAMPLE 2 — SLIGHTLY SUPERHEATED STEAM FROM SUPERHEATER INLET HEADER.
SAMPLE 3 — SUPERHEATED STEAM AFTER SUPERHEATER FIRST PASS.
SAMPLE 4 — SUPERHEATED STEAM AFTER SUPERHEATER SECOND PASS.
SAMPLE 5 — SUPERHEATED STEAM FROM BOILER STEAM LINE.
SAMPLE 6 — SUPERHEATED STEAM AT TURBINE INLET.
SAMPLE 7 — SLIGHTLY SUPERHEATED STEAM FROM TURBINE OUTLET.

FIG. 1.—Sampling Locations in Boiler-Turbine System.

pressure of 600 psi and interconnected through the plant main steam line.

A typical sodium analysis of these samples is shown on Fig. 2. Although the sodium content of the saturated steam sample was 240 ppb, the concentration of sodium in all superheated steam samples was in the order of 10 ppb. The difference between the measured sodium values in the saturated and superheated steam samples was so large that the validity of the results was questionable. A comprehensive analysis of the data and conditions of the test led to the following three possible explanations for the unexpected test results:

1. The simplest and most evident explanation was that a large percentage of the impurities in the steam was forming a deposit in the superheater supply tubes. However, the rate of deposition required to remove this quantity of impurities from the steam would have led to severe pluggage in a short time, but visual inspection of these tubes showed them to be completely free of deposits.

2. As mentioned previously, the task of obtaining a truly representative sample of saturated steam is quite difficult; therefore, the noted discrepancy may be due to the lack of representative sampling. However, although it is doubtful that the type of sampling nozzle used to collect the saturated steam sample gave a representative sample, it is also doubtful that the error involved could account for the large discrepancy noted.

3. It was observed, when the superheated steam sampling lines were shut off for a period of time, that the impurity content of the condensate formed in the lines was extremely high. This effect was not observed in the saturated steam sampling line. This observation led to the suspicion that the impurities in the superheated steam samples were forming deposits in the sampling lines, and, consequently, the sample reaching the measuring instrument contained only a fraction of the amount of impurities in the steam that was sampled. This explanation is supported further by the observations of others who have frequently reported that superheated steam sampling lines often become plugged when operated continuously.

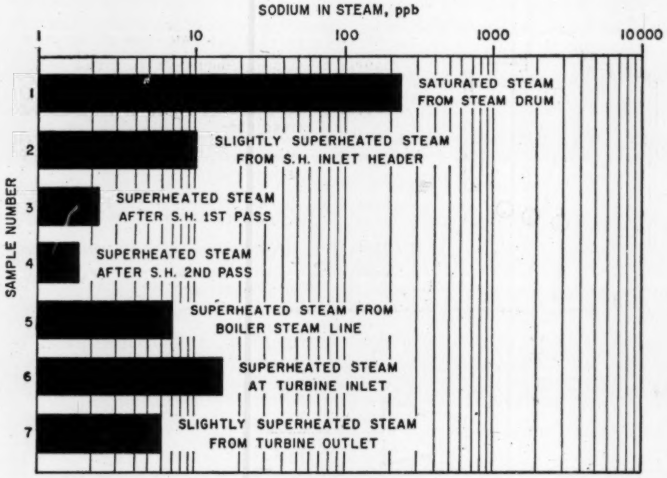

FIG. 2.—Typical Sodium Concentrations for Boiler-Turbine System Steam Samples.

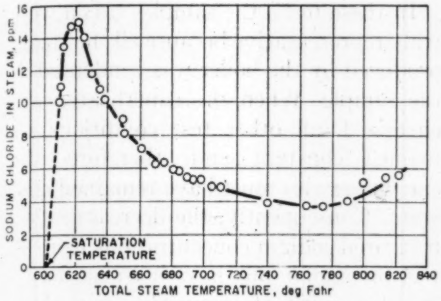

FIG. 3.—Solubility of Sodium Chloride in Superheated Steam at 1545 psi Pressure.

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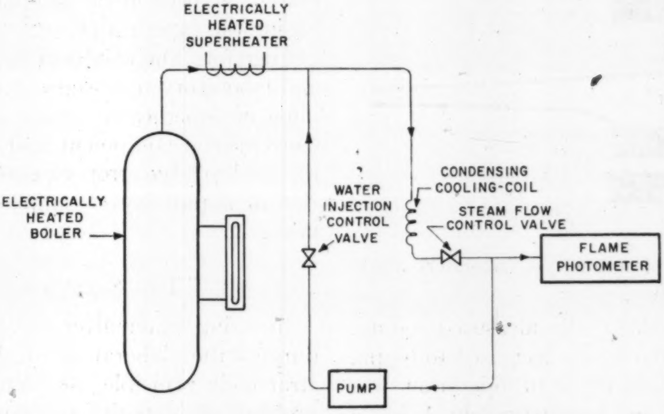
ELECTRICALLY HEATED SUPERHEATER
ELECTRICALLY HEATED BOILER
WATER INJECTION CONTROL VALVE
CONDENSING COOLING-COIL
STEAM FLOW CONTROL VALVE
FLAME PHOTOMETER
PUMP

FIG. 4.—Schematic Diagram of Laboratory Apparatus.

Although the evidence seemed to indicate that impurities in superheated steam samples tend to form deposits on sampling line surfaces, the reason for such deposition was not readily apparent. The most plausible explanation seemed to be that the impurities in superheated steam are present in vaporous form, and the steam becomes supersaturated with these materials as the temperature and pressure is reduced. Since the greatest temperature reduction occurs at the surface of the sampling line, the impurities tend to precipitate on the sampling line surfaces as solid deposits. This mechanism would be analogous to silica deposition in steam turbines. The data of Straub (4) seem to support this theory. Figure 3 shows the solubility of sodium chloride in superheated steam at 1545 psi, reproduced from Straub's data. The curve has been extended down to saturation temperature to illustrate the relatively large concentrations of sodium chloride that probably can be dissolved in steam with only a few degrees of superheat, and also that the solubility reaches a very small value as saturation temperature is approached. The work of Straub and others shows that other boiler water constituents have similar solubility characteristics, although the actual magnitude of the solubility may vary greatly for different constituents.

Such a theory cannot be investigated conveniently in the field, so a simple laboratory apparatus was constructed for this purpose (Fig. 4). It consists of a small electrically-heated, natural-separation boiler; an electrically-heated superheater; a means for injecting water after the superheater; a condensing-cooling coil; a throttling valve; and a continuously recording flame photometer. The boiler was operated at 600 psig. The solids concentration in the boiler water was held relatively constant at 4000 ppm with a pH of approximately 11. Sodium chloride represented about one-half of the total solids.

The results of a typical test run are shown on Fig. 5. The throttling valve was adjusted to provide a steam flow sufficient to produce a measurable amount of carry-over from the boiler. When the superheater was not used, the sodium content of the steam was 90 ppb. After the superheater was energized and adjusted to provide approximately 10 deg of superheat, the measured sodium content of the steam decreased to 6 ppb. After this condition of operation was maintained for approximately 1 hr, a small portion of the condensed sample was injected into the system immediately after the superheater. The amount of water injected was slightly more than that required to remove the superheat. The measured sodium content rose momentarily to an estimated value of 100 ppm and then slowly decreased to 85 ppb. When the superheater was turned off and the water injection discontinued, the measured sodium concentration returned to the 90 ppb value. (Maintaining perfectly stabilized conditions with such a small-volume system on manual control is nearly impossible and no significance should be attached to the difference between 85 and 90 ppb.)

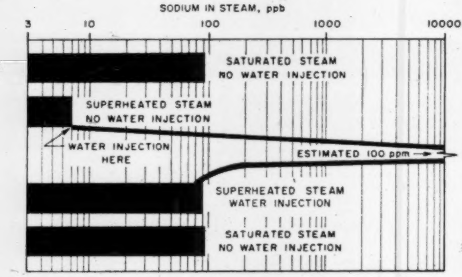

FIG. 5.—Typical Results of Laboratory Steam Sampling Test.

In these tests, the samples taken were truly representative because all the steam produced by the boiler was condensed as the sample. When the superheater was energized, all other test conditions remained constant and, therefore, the carry-over also must have remained constant. Consequently, the decrease in the measured sodium concentrations from 90 to 6 ppb could only have been caused by the deposition of impurities somewhere in the system. Since in the laboratory tests the water was injected after the superheater, the impurities redissolved by the injected water could not have been deposited in the superheater but must have been in the sampling line following the superheater.

Therefore, the results of both the field and laboratory tests show that the sampling of superheated steam when using conventional equipment and techniques can lead to large errors caused by deposition of impurities on sampling line surfaces.

THE SOLUTION

Injecting water after the superheater during the laboratory tests demonstrated, in principle, the solution to the problem of deposits on sampling line surfaces. If the superheated steam is contacted with a sufficient amount of water to remove the superheat before the sample is reduced in pressure or comes in contact with a cooler surface, the impurities will remain in the sample. Further, if a portion of the condensed sample is used for injection, the impurity content of the sample is not altered, and no correction is required.

The practical application of this principle requires that the superheat must be removed from the sample in the sampling nozzle itself, for cooler surfaces always exist external to the steam line. A type of sampling nozzle utilizing this principle is shown on Fig. 6. This arrangement consists of a standard steam sampling nozzle with a tube inserted so that its open end extends beyond the last sampling port. Separate external connections are provided for the internal tube and for the annulus surrounding the internal tube. When the nozzle is installed in the top of a horizontal steam line or in a vertical steam line with downward flow, water is injected through the annulus. As the steam sample enters the sampling ports, it is desuperheated by contact with the water and the desuperheated steam sample is removed through the central tube. If the nozzle is inserted through the bottom of a horizontal steam line, the flow direction is reversed, that is, the water is injected through the central tube and the sample is desuperheated and removed from the annulus. This type of nozzle should not be used in a vertical steam line with upward flow, for the injected water may drain from the nozzle through the sampling ports.

Figures 7 and 8 show two arrangements that may be used to inject a portion of the condensed sample into the nozzle. Figure 7 shows a natural circulation arrangement in which the density difference between water and a steam-water mixture is utilized to provide the pressure necessary to inject the required amount of water into the nozzle. The arrangement illustrated in Fig. 8 includes a pump to effect water injection.

Surprisingly, the first application of this principle involved the sampling of saturated steam. An industrial boiler operating at 250 psig pressure in a chemical plant required flushing of deposits from the superheater during semi-annual outages and occasionally suffered the failure of a superheater tube because of deposits. The standard dry-pan sample, which provides an average sample over the length of the steam drum, showed no measurable carry-over. Therefore, individual standard sampling nozzles were installed in 6 of the 20 superheater supply tubes, spaced approximately equally over the length of the drum. Samples taken from these locations also failed to show any evidence of carry-over, even when the sodium tracer technique was used. However, when the sampling lines were shut off and allowed to fill with condensate, it was noticed that the sodium concentration of the condensate was very high.

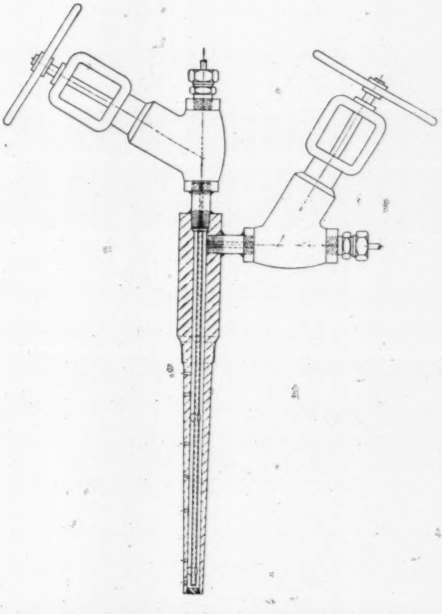

FIG. 6.—Improved Design Nozzle for Sampling Superheated Steam.

The design of this boiler was such that the sampling nozzles and parts of the sampling lines were inside the boiler casing and, thus, were surrounded by hot gases. Also, the sampling lines were $\frac{1}{4}$-inch stainless steel tubing with a small bore, and the sampling rate of approximately 100 lb per hr was sufficient to cause an appreciable pressure drop in the lines. It was reasoned that the steam may have become superheated in the sampling lines. At the relatively low pressure of 250 psi, the solubility of impurities in superheated steam would be very limited, and, consequently, the impurities could be deposited in the sampling lines.

Because of these circumstances, the standard sampling nozzles were replaced with the new-type nozzles. In this case, enough of the condensed sample was injected into the saturated steam to prevent its becoming superheated as it flowed through the sampling lines.

The use of the new-type sampling nozzles showed considerably higher carry-over at high loads than did the standard nozzle. Fig. 9 shows the results obtained from the sampling point that indicated the highest carry-over of the six sampling points. Whereas the standard nozzle had shown sodium concentrations less than 0.1 ppb for all conditions, the new-type nozzle showed increasing carry-over from 0.1 ppb sodium at 70 per cent load to about 40 ppb sodium at full load. The 40 ppb sodium

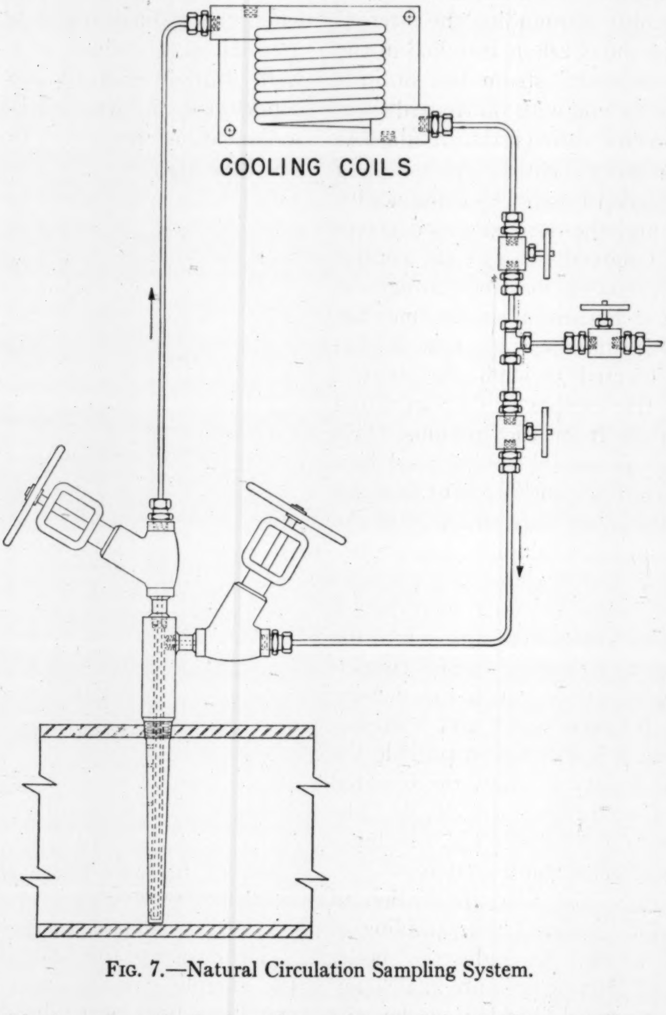

FIG. 7.—Natural Circulation Sampling System.

would correspond to about 0.12 ppm total solids. Although this amount of carry-over is well below the guaranteed value of 1 ppm, and the average carry-over from the boiler was considerably less than that indicated by the sampling point selected, the carry-over was sufficient to cause superheater deposits at this low pressure, just as it was sufficient to cause deposits in the sampling lines. Subsequently, this small amount of carry-over was reduced by the installation of additional separating equipment, and the superheater deposit problem was eliminated.

Figure 10 shows a recording of the sodium content from the same sampling point and illustrates the characteristics of the deposit problem. When no water was injected, the measured sodium concentration was less than 0.1 ppb. Although the steam as sampled contained appreciably more than 0.1 ppb, most of the impurities were being deposited on the sampling line surfaces and not reaching the measuring instrument. When water was injected, the accumulated deposit was dissolved, as indicated by the fact that the sodium concentration exceeded 100 ppb momentarily. After the accumulated deposit was removed, the sodium concentration of the sample stabilized at the true value of 2 ppb.

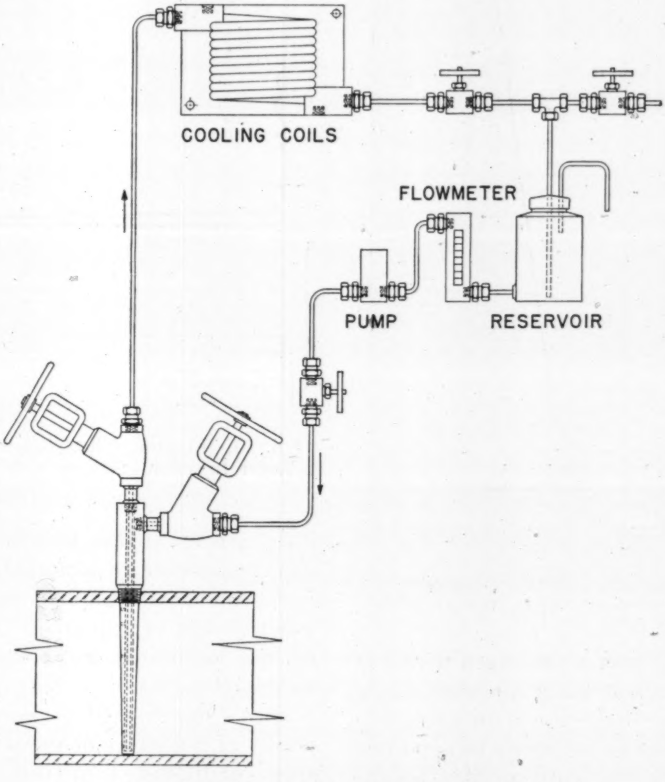

FIG. 8.—Forced Injection Sampling System.

Nozzles of this type were installed in two boilers operating at 1300 psig to determine if the submerged attemperator bundles were leaking. Although the impurity level was so low that no detectable differences could be noted with and without water injection (thereby indicating that no attemperator leakage was occurring), a slight momentary rise in sodium content was observed when water was injected after a prolonged period of sampling without water injection. These results show that even with extremely pure steam, impurities may be deposited on sampling line surfaces if standard sampling techniques and equipment are used.

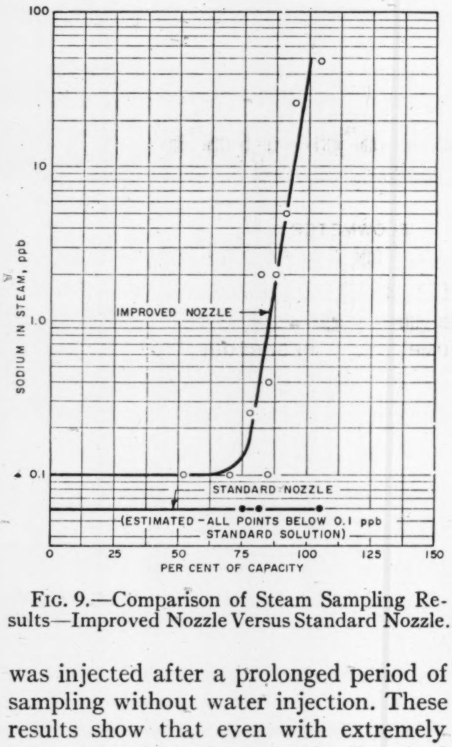

Fig. 9.—Comparison of Steam Sampling Results—Improved Nozzle Versus Standard Nozzle.

Summary

The sampling of superheated steam (and saturated steam under certain conditions) with conventional sampling equipment and techniques can result in very large errors caused by impurities depositing on sampling line surfaces. This deposition is apparently caused by the decreasing ability of superheated steam to retain impurities in vaporous form as steam temperature is reduced. The errors resulting from deposition can be avoided by using a special sampling nozzle into which a sufficient amount of water is injected to remove all superheat from the steam sample. If a portion of the condensed sample is injected, the impurity concentration of the sample will not be altered and no correction is necessary.

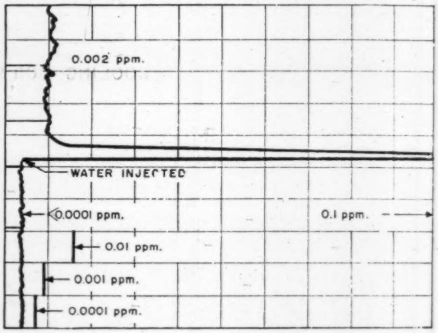

Fig. 10.—Recorder Chart Illustrating Effect of Water Injection—Sodium in Steam.

Since superheated steam consists usually of a mixture of vapors or, under some conditions, a mixture of vapors containing extremely fine dust, a representative sample can be obtained. The technique and equipment described herein must be employed to assure that the representative sample collected will be delivered to the measuring instrument.

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IMPURITIES IN STEAM FROM HIGH-PRESSURE BOILERS

By R. C. Ulmer[1] and H. A. Klein[2]

Synopsis

A research program was undertaken to determine the extent to which sodium salts are volatilized under the conditions existing in high-pressure boilers and to establish how vaporous carry-over might influence the measurement and control of steam purity. Field tests were performed on three boilers operating in the 2500 to 2600-psi range. Routine measurements of total carry-over were performed, and a special sampling system was installed at the test sites to permit direct determinations of vaporous carry-over. Particular emphasis was placed in the study of sodium chloride characteristics, although other salts were evaluated to a lesser degree.

The results obtained indicate that the vaporous carry-over of sodium chloride is relatively low in this pressure range as long as the salt concentration in the boiler water is maintained within reasonable limits. The magnitude of vaporous carry-over obtained in the field tests was appreciably less than that obtained by other investigators in laboratory model tests. Possible explanations for this discrepancy are discussed.

It was concluded that vaporous carry-over of sodium salts from boilers operating at 2600 psi or less will not present a serious problem with regard to turbine deposition as long as the total salt concentration in the boiler water is maintained below 25 ppm. No attempt was made to extrapolate the data obtained to predict characteristics at higher pressures. It was recognized that the vaporous carry-over of sodium salts would complicate the determination of high-pressure steam purity.

From the standpoint of the operator whose turbine is being fouled with deposits, it makes little difference whether the solids present in his steam are the result of mechanical carry-over or vaporous carry-over. The mechanism of solids transport is extremely important, however, to the chemist or engineer who is trying to measure and control steam purity.

For the purposes of this paper, "solids" refers to all materials which are solid in the free state at ambient temperatures. The term "solids" may be applied even when such materials are in liquid solution or in the vapor phase. Mechanical carry-over refers to the presence of solids in the steam resulting from the entrainment of boiler water with the steam. Vaporous or volatile carry-over refers to the presence of solids in the steam due to volatilization of some component in the boiler water. Total carry-over refers to the sum of the mechanical plus the vaporous carry-over.

Mechanical carry-over is a function of numerous factors including steaming rate, water-level, boiler design, and the foaming characteristics of the boiler water. In general, mechanical carry-over would be expected to be nonselective with respect to individual constituents since the entrained boiler water presumably contains the same ratio of constituents as the boiler water itself. Under conditions of pure mechanical carry-over, the determination of a single constituent in the boiler water and steam will determine the per cent moisture content and the ratio of all other components present.

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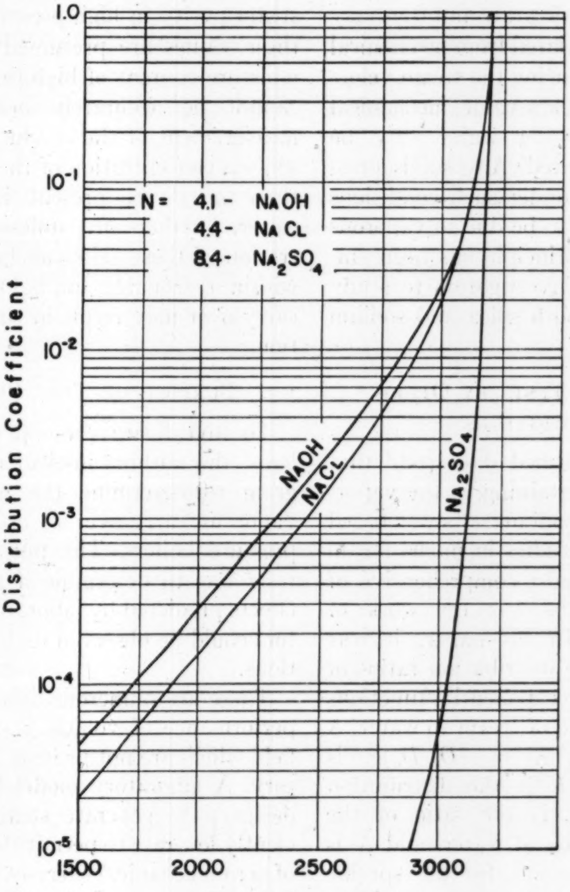

FIG. 1.—Distribution Coefficients of Salts from Equation of Styrikovich:

$$K = \left(\frac{D_s}{D_w}\right)^N$$

Vaporous carry-over, on the other hand, is completely selective in nature since it is dependent upon the volatility of a specific constituent under observation. Theoretically, for a given operating pressure, the ratio of the concentration of the vaporous component in the steam to its concentration in the boiler water is constant. This constant coefficient is referred to as the distribution ratio. The ions present in the boiler water are not necessarily volatilized as such, and some intermediate product may be involved in the vaporization process. Arbitrarily determined ratios may not be real distribution ratios, and caution must be employed in interpreting results.

Vaporous carry-over is, however, independent of steaming rate and thus may be readily differentiated from mechanical carry-over. By reducing the steam velocity to a sufficiently low value, mechanical entrainment of boiler water may be completely eliminated. Any solids present in the steam under such conditions may be presumed to be due to vaporous carry-over. This principle has been employed by many investigators to study the volatility of both silica and sodium salts.

LABORATORY TESTS BY OTHER INVESTIGATORS

All of the published data (**1-5**)[3] that could be found pertaining to the vaporous carry-over of sodium salts was based upon laboratory studies in model steam generators. The most comprehensive of these investigations was the work of Styrikovich (**1**). In his paper, it was theorized that the distribution ratios of all compounds are exponential functions of the density ratio of steam to water. A general equation, $K = (D_s/D_w)^N$, is presented where K is the distribution coefficient, D_s/D_w is the ratio of the densities of steam and water, and N is the volatility constant for the specific compound. Coulter et al (**2**) observed this same approximate relationship with regard to the distribution ratios for silica. Figure 1 is a plot of the distribution coefficients of three sodium salts as a function of pressure based on the data of Styrikovich. On the basis of this plot, sodium hydroxide and sodium chloride volatilize to a significant degree at 2500 psi and above. Only at very high pressures would the vaporous carry-over of sodium sulfate be of any serious concern.

The distribution coefficients for sodium hydroxide and sodium chloride presented in Fig. 1 are sufficiently high to affect both the measurement and control of steam purity in high-pressure boilers. If these values are presumed correct, the moisture content of high-pressure steam cannot be accurately determined by measurement of the conductivity or the sodium concentration of the steam when these salts[4] are present in the boiler water. Furthermore, unless the concentration of these salts can be maintained within reasonable limits, their volatile carry-over may result in turbine deposition.

PURPOSE OF TEST PROGRAM

To investigate these potential problems, the authors instituted a test program to determine the magnitude of vaporous carry-over in operating high-pressure boilers. The purpose of these tests was to determine if the volatility effects predicted by laboratory investigators could be observed under field conditions.

There are numerous difficulties in the performance of volatility studies in the field which are not present in the laboratory. A laboratory model boiler can be designed to generate steam at a sufficiently low rate to permit the assumption of zero mechanical carry-over. An expensive high-pressure boiler is rarely, if ever, operated at a sufficiently light load to ensure this assumption. Furthermore, boiler water concentrations can be arbitrarily established in the laboratory to produce concentrations in the steam which can be analyzed by standard methods, and radioactive tracers may be effectively employed to greatly improve analytical precision. For obvious reasons, neither salts nor radioisotopes can be indiscriminantly added to a utility boiler. However, conditions in laboratory tests never really simulate conditions in the operating boiler. For this reason, the field test program was considered necessary to determine if the laboratory data could be applied to boiler operation.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

[4] For uniform terminology, sodium hydroxide will be referred to as a salt.

TEST SITES

The vaporous carry-over test program was performed on three high-pressure boilers belonging respectively to three different utilities. The first unit tested was the No. 4 boiler at the W. C. Beckjord Power Station of the Cincinnati Gas and Electric Co. At full load, this boiler produces about 1,200,000 lb per hr of steam. The second test site was the No. 4 boiler at the Chesterfield Power Station of the Virginia Electric and Power Co. which produces 1,300,000 lb per hr of steam at full load. The third boiler tested was the No. 5 boiler of the Ashtabula Power Station of the Cleveland Electric Illuminating Co. The full-load steaming rate of this unit is 1,600,000 lb per hr. All three of these boilers are controlled-circulation units with operating drum pressures of from 2500 to 2600 psig. For brevity in the text, the three test facilities may be referred to respectively as Beckjord, Chesterfield, and Ashtabula.

Although the boiler water specifications varied somewhat, the normal control in all three boilers may be described as a low solids treatment with the salt content in the boiler water generally maintained below 25 ppm. Slightly increased salt concentrations were permitted during the test periods to provide for more accurate analyses.

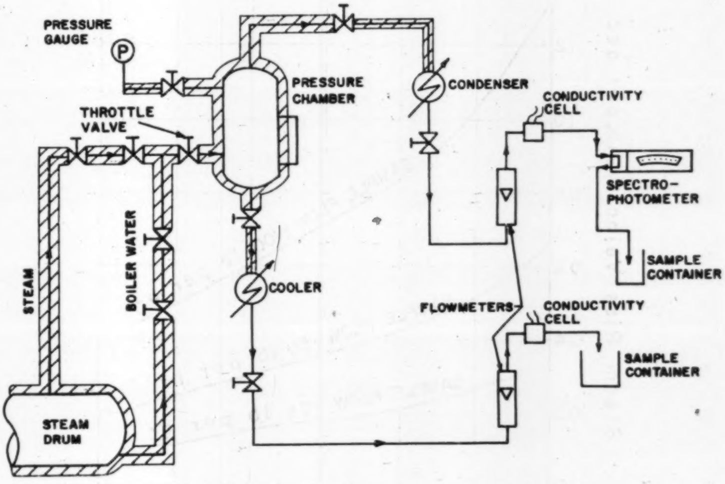

FIG. 2.—Sampling System for Vaporous Carry-Over Tests.

TEST PROCEDURE

Because of the relatively low solids content in the boiler water, sodium determinations were used as the basis for all carry-over measurements. The instrument employed for analysis was a Beckman Model B flame spectrophotometer equipped to permit continuous sample analysis. Complete analyses were performed on most boiler water samples. Sodium was the only determination performed routinely on steam samples; however, a number of steam samples were concentrated to permit the analysis of other constituents and to check low-range sodium determinations.

Total carry-over was determined by continuous sodium analysis of the flowing steam sample over a period of about an hour. Since total carry-over must always be equal to or greater than the vaporous carry-over, these measurements established maximum limit for vaporous carry-over at the operating pressures of the respective boilers.

A special sampling system was designed to permit the determination of vaporous carry-over, as such, at various pressures. This system is shown schematically in Fig. 2. Samples of steam and boiler water were simultaneously introduced into the pressure chamber at controlled rates. The mixture of steam and boiler water was then separated in the chamber with the water being discharged at the bottom and the steam at the top. The pressure in the chamber could be maintained at drum pressure by taking all of the pressure drop across the discharge valves, or the pressure could be reduced to any desired lower pressure by taking a portion of the pressure drop across the inlet throttle valve. Effective control of pressure and flow was possible with the system as shown.

The effectiveness of separation is dependent upon the maintenance of a low steam-rise-velocity through the pressure chamber. This velocity is a function of both sampling rate and steam pressure. The relationship is illustrated in Fig. 3. No detectable mechanical carry-over resulted within the pressure ranges tested with steam sample flows as high as 150 lb per hr. For convenience, however, most tests were performed with steam flows of from 25 to 50 lb per hr. In general, boiler water and steam flows were approximately equal.

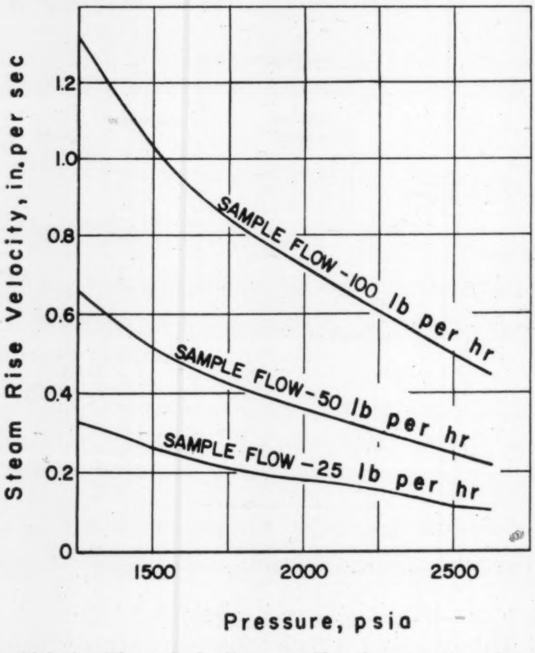

FIG. 3.—Steam Velocity Through the Pressure Chamber as a Function of Pressure.

Water level control in the pressure chamber was critical in the performance of these tests. A special method of water level control was integrated into the design of the pressure chamber. Figure 4 is a sketch of the pressure chamber used in the tests performed at the Chesterfield and Ashtabula stations. Water level in the chamber was reflected by subcooling in the external water column. Thermocouple points which became submerged indicated temperature reductions of from 30 to 70 F. Thermocouple measurements were continuously monitored on a temperature recorder which permitted excellent control of water level. In the initial tests at Beckjord, a somewhat shorter pressure chamber with only three thermocouple points was used, and temperature measurements were made manually with a potentiometer. With this system, level control was not completely adequate, and mechanical carry-over was a problem on several occasions. For this reason, the chamber was redesigned for the subsequent tests. In general, water level was controlled between the first and second thermocouple points, although mechanical carry-over did not occur at normal steam flows unless the entire water column became submerged.

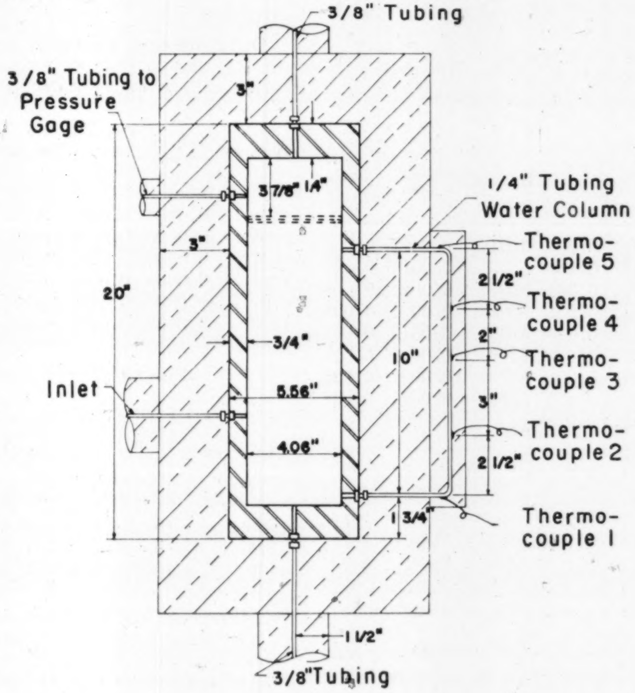

FIG. 4.—Pressure Chamber for Vaporous Carry-Over Tests.

Considerable effort was taken to make the entire sampling system adiabatic. The inlet steam and boiler water lines were made as short as possible, and all surfaces from the boiler to the discharge valves were heavily insulated. Heat loss in the inlet lines is not particularly serious; however, precondensation will tend to dilute the boiler water in the pressure chamber. Condensation in the chamber itself is critical since postcondensation may establish a second water-steam equilibrium in the upper part of the vessel. This washing effect can reduce the apparent vaporous carry-over. Every effort was made to prevent condensation in the pressure chamber by insulation. At Ashtabula the vessel was steam jacketed and then covered with insulation. The steam jacket was actually a tightly-wound coil supplied with steam at the same pressure as in the chamber. The possible effects of postcondensation on the test results will be discussed subsequently.

TABLE I.—TOTAL CARRY-OVER TESTS AT THE CHESTERFIELD POWER STATION.

Drum pressure, 2600 psig.
Load, 170 Mw.

STEAM SAMPLES

Time, pm	Sodium, ppm	Carry-over, per cent	Time, pm	Sodium, ppm	Carry-over, per cent
4:46....	0.0027	0.036	5:04...	0.0021	0.028
4:48....	0.0027	0.036	5:05...	0.0019	0.026
4:50....	0.0023	0.031	5:07...	0.0021	0.028
4:52....	0.0021	0.028	5:08...	0.0021	0.028
4:55....	0.0018	0.024	5:09...	0.0019	0.026
4:56....	0.0021	0.028	5:10...	0.0019	0.026
4:57....	0.0021	0.028	5:12...	0.0021	0.028
5:00....	0.0018	0.024	5:15...	0.0021	0.028
5:02....	0.0021	0.028			

BOILER WATER SAMPLES

4:46....	7.5	5.7	5:02...	7.4	5.7
4:52....	7.5	5.7	5:07...	7.4	5.7
4:57....	7.5	5.7	5:15...	7.5	5.7

Average sodium in stream = 0.0021 ppm.
Average sodium in boiler water = 7.5 ppm.
Average total carry-over = 0.028 per cent.
Average sodium chloride in boiler water = 9.4 ppm.
Maximum possible distribution ratio for sodium chloride = 5.7×10^{-4}.

For the final tests which were performed at Ashtabula, a small pressure-pot was installed in the sampling system to permit injection of salts directly into the pressure chamber. The purpose of this was to study the equilibrium between steam and concentrated boiler water. A concentrated salt solution was first injected into the steam-filled pressure chamber, and then high-pressure steam was introduced into the vessel below the water level and was discharged at the top. Once equilibrium was obtained, level was maintained constant. A small amount of condensate was introduced with the steam, and an equal amount of water was continuously discharged from the bottom of the vessel. This resulted in a gradual dilution of the boiler water but, at the same time, provided a sample for analysis. Both the steam and boiler water were continuously monitored over a wide range of concentrations. Concentrated solutions of sodium chloride and sodium sulfate were evaluated in this manner.

TABLE II.—TOTAL CARRY-OVER TESTS AT THE ASHTABULA POWER STATION.

Drum pressure, 2500 psig.

STEAM SAMPLES

Time, pm	Sodium, ppm	Carry-over, per cent	Time, pm	Sodium, ppm	Carry-over, per cent
1:02....	0.0034	0.032	1:11...	0.0028	0.026
1:05....	0.0026	0.025	1:13...	0.0026	0.025
1:07....	0.0030	0.028	1:15...	0.0025	0.024
1:08....	0.0035	0.033	1:17...	0.0026	0.025
1:09....	0.0030	0.028	1:18...	0.0026	0.025
1:10....	0.0032	0.030	1:25...	0.0026	0.025

BOILER WATER SAMPLES

Time, pm	Sodium, ppm	Chloride, ppm	Phosphate, ppm	Conductivity, micromhos	pH
1:00......	10.6	9.3	1.1	64.2	10.0
1:15......	10.6	9.3	1.0	66.0	10.0
1:20......	10.6	9.1	1.1	68.5	10.0

Average sodium in steam = 0.0029 ppm.
Average sodium in boiler water = 10.6 ppm.
Average total carry-over = 0.027 per cent.
Average sodium chloride in boiler water = 15.3 ppm.
Maximum possible distribution ratio for sodium chloride = 4.7×10^{-4}.

Test Results

Tables I and II present the total carry-over test data obtained at the Chesterfield and Ashtabula stations. The data were obtained by continuous sodium analysis of the regular steam sample. Boiler water samples were intermittently collected for analysis. Tests of this type were not performed at Beckjord; however, spot measurements of total carry-over indicated the same approximate relationship.

It may be noted that some sodium values have been reported to the nearest tenth of a part per billion. The authors do not presume the test to be accurate to this degree; however, it was believed desirable to report the data as closely as they could be read from the calibration curve.

TABLE III.—TECHNIQUES FOR CONCENTRATING STEAM SPECIMENS.

Station	Pressure, psi	Steam				Boiler Water		
		Direct Sodium Measurement, ppm	Method of Concentration	Number of Concentrations	Sodium in Concentrate, ppm	Sodium, ppm	Phosphate, ppm	Chloride, ppm
Beckjord	2550	0.0010	Expansion	10	0.0130	2.6	...	...
Beckjord	2550	0.0010	Expansion	5	0.0055	2.6	...	...
Chesterfield	2575	0.0014	Evaporation	27.2	0.0392	8.7	1.2	6.6
Chesterfield	2375	0.0013	Evaporation	35.2	0.0440	8.4	1.2	6.6
Ashtabula	2450	0.0021	Evaporation	34.2	0.0730	8.0	3.0	6.6
Ashtabula	2550	0.0018	Evaporation	43.1	0.0710	9.4	2.2	7.7

Because the sodium concentrations in the steam were always in the low part-per-billion range, methods for concentrating steam samples were occasionally employed to confirm the test results. One of the methods employed was that proposed by Gurney (6). In this procedure, the high-pressure steam sample was throttled to a lower pressure, which condensed a percentage of the steam. This condensate absorbed virtually all of the solids originally present in the steam. By separating this moisture from the steam in the pressure chamber, the solids present in the steam could be concentrated by some factor dependent upon the percentage of moisture formed. The advantage of this technique was that it was quite rapid and permitted direct-flow analysis. A second concentration procedure employed a "continuous sample evaporator." This equipment permitted the evaporation (without boiling) of a large steam sample to a relatively small volume which could then be analyzed as desired. With this technique, virtually any number of concentrations could be obtained; however, the process was more time consuming than the expansion procedure. Both concentration methods showed good agreement with the direct sodium analyses. Some representative values are presented in Table III.

Of primary interest in the test program were the direct determinations of vaporous carry-over under typical boiler water conditions. However, to ensure that the boiler water would contain one of the two salts predicted to have a significant volatility, the stations involved were requested to introduce a small amount of sodium chloride into the boilers to be tested. Sodium chloride was selected over sodium hydroxide because of possible side effects associated with

TABLE IV.—VAPOROUS CARRY-OVER TESTS.

Pressure, psig		Sodium, ppm	Chloride, ppm	Phosphate, ppm	Conductivity, micromhos	$K = \frac{\text{Na(Steam)}}{\text{Na(Boiler Water)}}$
BECKJORD POWER STATION						
2500	Steam	0.0010	...	...	2.80	1.4×10^{-4}
	Water	7.2	9.2	...	35.0	
2000	Steam	0.0007	...	...	2.75	6.9×10^{-5}
	Water	10.2	13.1	...	49.6	
1500	Steam	0.0006	...	...	2.80	5.2×10^{-5}
	Water	11.6	16.2	...	57.0	
2500	Steam	0.0020	...	...	1.45	2.0×10^{-4}
	Water	10.0	13.1	...	41.6	
2500	Steam	0.0008	...	...	2.02	7.1×10^{-5}
	Water	11.2	13.8	...	41.0	
1950	Steam	0.0007	...	...	2.10	6.3×10^{-5}
	Water	11.2	13.5	...	38.9	
1450	Steam	0.0005	...	...	2.00	2.8×10^{-5}
	Water	18.0	21.50	...	86.5	
CHESTERFIELD POWER STATION						
2600	Steam	0.0013	...	...	2.54	2.5×10^{-4}
	Water	5.2	...	2.2	35.1	
2400	Steam	0.0009	...	...	2.45	1.8×10^{-4}
	Water	5.0	...	2.1	36.0	
2200	Steam	0.0007	...	...	2.60	1.4×10^{-4}
	Water	5.2	...	2.0	40.4	
2025	Steam	0.0005	...	...	2.46	9.6×10^{-5}
	Water	5.2	...	2.0	37.0	
1800	Steam	0.0003	...	...	2.35	5.4×10^{-5}
	Water	5.6	...	2.0	35.4	
1600	Steam	0.0003	...	...	2.55	5.7×10^{-5}
	Water	5.3	...	2.0	36.9	
2475	Steam	0.0010	...	...	2.50	1.3×10^{-4}
	Water	7.6	...	6.5	52.8	
2175	Steam	0.0009	...	...	2.07	1.2×10^{-4}
	Water	7.8	...	6.0	59.8	
1975	Steam	0.0007	...	...	2.25	9.0×10^{-5}
	Water	7.8	...	6.0	60.2	
1800	Steam	0.0007	...	...	2.26	9.0×10^{-5}
	Water	7.8	...	6.2	59.8	
1600	Steam	0.0006	...	...	2.32	8.0×10^{-5}
	Water	7.5	...	6.35	58.5	
2575	Steam	0.0014	...	...	1.86	1.6×10^{-4}
	Water	8.7	6.6	1.23	51.8	
2375	Steam	0.0013	...	...	2.14	1.5×10^{-4}
	Water	8.4	6.6	1.23	51.9	

TABLE IV.—*Continued*

Pressure, psig		Sodium, ppm	Chloride, ppm	Phosphate, ppm	Conductivity, micromhos	$K = \frac{\text{Na(Steam)}}{\text{Na(Boiler Water)}}$
CHESTERFIELD POWER STATION						
2225	Steam	0.0011	...	...	2.15	1.4×10^{-4}
	Water	8.0	6.1	1.75	56.0	
2000	Steam	0.0010	...	...	2.17	1.3×10^{-4}
	Water	8.0	6.3	1.24	55.5	
1800	Steam	0.0009	...	...	2.25	1.1×10^{-4}
	Water	8.2	6.6	1.24	55.6	
ASHTABULA POWER STATION						
2450	Steam	0.0021	...	...	2.14	2.6×10^{-4}
	Water	8.0	6.6	3.0	57.5	
2200	Steam	0.0019	...	...	2.10	1.9×10^{-4}
	Water	10.0	8.3	3.2	69.0	
1900	Steam	0.0019	...	...	2.20	1.9×10^{-4}
	Water	9.8	8.3	3.4	67.0	
1750	Steam	0.0015	...	...	2.15	1.6×10^{-4}
	Water	9.4	7.7	3.2	63.5	
1500	Steam	0.0015	...	...	2.20	1.5×10^{-4}
	Water	9.8	7.9	3.2	65.5	
2250	Steam	0.0019	...	...	1.68	2.0×10^{-4}
	Water	9.4	7.7	2.2	66.6	
2050	Steam	0.0012	...	...	1.67	1.4×10^{-4}
	Water	8.3	7.0	2.0	62.5	
1750	Steam	0.0015	...	...	1.67	1.9×10^{-4}
	Water	7.8	6.6	2.4	57.2	
1400	Steam	0.0010	...	...	1.64	1.3×10^{-4}
	Water	7.5	6.6	2.4	56.4	

the latter. Each of the three utilities consented to the request, and from 10 to 25 ppm of sodium chloride was maintained in the water in these boilers during the test period. Several test runs were performed before the addition of sodium chloride to the boilers for comparative purposes.

Each test run consisted of measurements with the chamber at full drum pressure and at a number of reduced pressures. The test range included roughly from 1500 to 2600 psig. Table IV presents a résumé of all the vaporous carry-over data collected at the three test sites.

Only at Ashtabula was the sampling system equipped to permit evaluation of vaporous carry-over from highly concentrated boiler water solutions. Sodium chloride and sodium sulfate were injected into the pressure chamber on separate test runs. After equilibrium was obtained, the steam and boiler water samples were continuously monitored over a wide range of concentrations. Tables V and VI present a résumé of the data obtained.

TABLE V.—SODIUM CHLORIDE INJECTION TEST, ASHTABULA POWER STATION—UNIT NO. 5, PRESSURE 2550 psig.

Time	Boiler Water		Steam		$K \times 10^{-4}$
	Conductivity, micromhos	Sodium, ppm	Conductivity, micromhos	Sodium, ppm	
6:51	6600	1590	...	...	...
6:53	5550	1330	...	...	...
6:54	5040	1290	...	...	...
6:55	4850	1120	7.11	0.800	7.14
7:04	2100	400	5.9	0.400	10.0
7:05	2000	380	5.11	0.300	7.89
7:06	1780	340	4.71	...	...
7:07	1650	310	2.74	0.175	5.65
7:08	1540	290	2.66	0.140	4.83
7:09	1450	270	2.51	0.125	4.63
7:10	1320	245	2.45	0.106	4.32
7:11	1130	210	2.9	0.096	4.57
7:13	935	183	2.81	0.088	4.81
7:14	836	163	2.25	0.075	4.60
7:15	770	150	2.16	0.060	4.00
7:16	701	138	2.1	0.047	3.41
7:17	638	125	2.06	0.037	2.96
7:18	583	113	2.04	0.031	2.74
7:19	540	105	2.02	0.027	2.57
7:20	496	97	1.99	0.022	2.27
7:21	452	87	1.99	0.021	2.41
7:22	405	79	1.95	0.022	2.78
7:23	353	69	1.95	0.019	2.75
7:24	322	62	1.95	0.014	2.26
7:25	300	58	1.94	0.017	2.93
7:26	275	55	1.91	0.014	2.55
7:27	264	53	1.91	0.011	2.08
7:28	242	48	1.89	0.0089	1.85
7:29	224	43	1.88	0.0065	1.51
7:30	205	39	1.88	0.0060	1.54
7:31	192	37	1.88	0.0050	1.35
7:32	169	33	1.89	0.0040	1.21
7:33	155	30	1.90	0.0035	1.17
7:34	140	26	1.90	0.0030	1.15
7:35	126	24	1.89	0.0030	1.25
7:36	117	22	1.86	0.0030	1.36
7:37	98.5	20	1.85	0.0025	1.25
7:38	90.3	18	1.86	0.0025	1.39
7:39	84.4	15	1.86	0.0023	1.53
7:40	78.0	14	1.86	0.0023	1.64
7:41	68.6	12	1.85	0.0023	1.91
7:42	61.5	12	1.86	0.0021	1.75
7:43	54.9	11	1.86	0.0020	1.82
7:44	49.2	10	1.86	0.0020	2.00
7:45	44.6	9	1.85	0.0018	2.00
7:46	39.7	8	1.84	0.0016	2.00
7:47	34.4	6	1.84	0.0015	2.50

DISCUSSION OF RESULTS

The results shown in Tables I and II indicate average total carry-over values of 0.028 per cent and 0.027 per cent for boilers operating at pressures of 2600 psig and 2550 psig, respectively. The limiting distribution ratios for sodium chloride were calculated by presuming that all of the carry-over was vaporous and that sodium chloride was the only volatile salt present. The values (5.7×10^{-4} and 4.7×10^{-4}) obtained were considerably less than those predicted using the equation of Styrikovich (Fig. 1).

TABLE VI.—SODIUM SULFATE INJECTION TEST, ASHTABULA POWER STATION—UNIT NO. 5.

Time	Boiler Water		Steam		$K \times 10^{-5}$
	Conductivity, micromhos	Sodium, ppm	Conductivity, micromhos	Sodium, ppm	
5:53	3400	810	...	0.058	7.16
5:54	3350	790	...	0.062	7.84
5:55	3400	810	...	0.060	7.41
5:56	3300	780	...	0.056	7.18
5:58	3200	760	...	0.056	7.37
5:59	3100	720	...	0.060	8.33
6:00	3000	700	...	0.053	7.57
6:02	2200	480	...	0.046	9.58
6:03	2100	460	...	0.043	9.35
6:04	1900	410	...	0.034	8.29
6:05	1800	380	...	0.034	8.95
6:06	1700	360	...	0.033	9.17
6:07	1600	340	...	0.034	10.00
6:08	1500	310	...	0.037	11.93
6:09	1450	300	...	0.034	11.33
6:10	1400	290	...	0.031	10.69
6:11	1300	265	...	0.030	11.32
6:12	1200	240	...	0.027	11.25
6:13	1150	215	...	0.025	11.63
6:14	1035	200	...	0.023	11.50
6:15	930	195	...	0.023	11.79
6:16	890	190	...	0.021	11.05
6:17	830	182	...	0.017	9.34
6:18	755	164	...	0.013	7.93
6:19	680	148	...	0.0125	8.45
6:20	615	132	...	0.0115	8.71
6.21	580	125	...	0.0100	8.00
6.22	550	120	...	0.0092	7.67
6.23	512	110	...	0.0077	7.00
6:24	500	108	...	0.0065	6.02

The direct determinations of vaporous carry-over (Table IV) produced even lower distribution ratios than those shown above. The sodium concentrations in the steam samples were all extremely low, and all distribution coefficients obtained were less than those predicted by other investigators. The possibility of postcondensation in the upper part of the pressure chamber was immediately suspected. Even a small amount of condensation can absorb a large percentage of the vaporized solids from steam when the distribution ratio is small. If a second water-steam equilibrium occurs in the upper part of the test vessel, the steam in contact with the condensate would be effectively washed. As a consequence, the apparent distribution ratio would be lower than the actual equilibrium value. The discrepancy between the two values is a function of the percentage condensation, the actual distribution ratio, and the effective contact between the steam and condensate. Figure 5 is an illustration of the potential error which can result from postcondensation in a test vessel. This plot assumes that all condensate is retained in the test vessel, and that the condensate absorbs solids to saturation. The plot, therefore, gives the maximum possible error which can be introduced by the washing phenomenon.

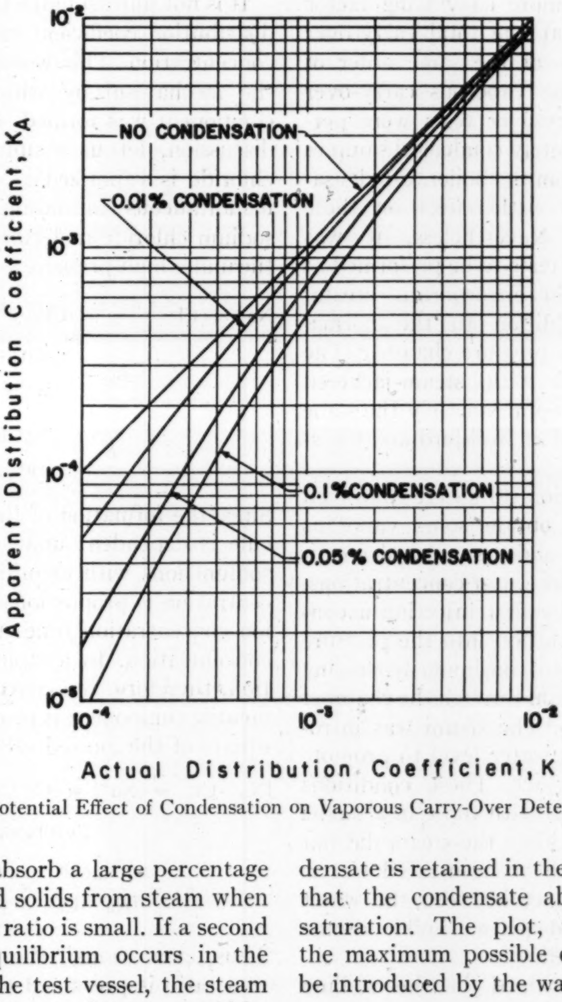

FIG. 5.—Potential Effect of Condensation on Vaporous Carry-Over Determinations.

Although heat losses can never be prevented completely by insulation alone, postcondensation was not believed to have been a problem at Beckjord or Chesterfield. The reason for this opinion was that the results were not affected by significant changes in steam flow through the pressure chamber. Presuming a constant rate of heat loss, an increased steam flow will reduce the percentage of condensate formed and should thus reduce the effective washing which could occur. A more convincing factor was the fact that the total carry-over test results were of the same order of magnitude as the vaporous carry-over data. Total carry-over tests were performed on completely condensed samples taken directly from the boiler. Condensation could have little effect on these determinations. Nevertheless, it was decided that the tests to be performed at Ashtabula would incorporate steam-jacketing in addition to the normal insulation on the pressure chamber. The results obtained with steam-jacketed equipment were approximately the same as those obtained at Beckjord and Chesterfield.

The salt injection tests were conceived in an effort to obtain some vaporous carry-over data under simulated conditions of high boiler water concentrations. This was done by first injecting a concentrated salt solution into the pressure chamber and then continuously passing high-pressure steam through the chamber and out the top. The steam was introduced below the water level to promote mixing and contact. These conditions were not identical with those in a model steam generator since the steam did not originate from the concentrated water; however, if contact between the water and steam is adequate, similar results might be expected.

The data obtained in the sodium chloride injection tests are rather interesting. The calculated distribution ratios are definitely higher with high salt concentrations in the water. This phenomenon may be peculiar to the type of test performed; however, it is noteworthy that the values obtained at the high concentrations are in fair agreement with the laboratory data of other investigators while the values obtained at low concentrations are in agreement with the coefficients we had previously obtained.

It is not unreasonable that a measured distribution coefficient might vary with concentration. This would depend upon the mechanism by which the volatile component was formed. For the sake of discussion, let us assume that sodium chloride is vaporized as a result of the instantaneous formation of a neutral sodium chloride molecule which is volatile under high-pressure boiler conditions:

$$\mathrm{Na}^+ + \mathrm{Cl}^- \xrightarrow{K_1} \mathrm{NaCl}^0_{(w)} \xrightarrow{K_2} \mathrm{NaCl}_{(s)} \quad (1)$$

$$K_1 = \frac{[\mathrm{NaCl}^0]_w}{[\mathrm{Na}^+][\mathrm{Cl}^-]} \quad (2)$$

$$K_2 = \frac{[\mathrm{NaCl}]_s}{[\mathrm{NaCl}]_w} \quad (3)$$

Since the formation of the neutral molecule is dependent upon the reaction of sodium ions with chloride ions, its concentration is proportional to the sodium ion concentration times the chloride ion concentration. In a stoichiometric solution, therefore, the concentration of the volatile component is proportional to the square of the ionized salt concentration:

$$[\mathrm{Na}^+\cdot\mathrm{Cl}^-] = [\mathrm{Na}^+] = [\mathrm{Cl}^-]$$

Stoichiometric solution . . (4)

$$K_1 = \frac{[\mathrm{NaCl}^0]}{[\mathrm{Na}^+][\mathrm{Cl}^-]} = \frac{[\mathrm{NaCl}^0]}{[\mathrm{Na}^+\cdot\mathrm{Cl}^-]^2} \quad (5)$$

Thus, even though the actual distribution ratio is presumed constant (Eq 3), the volatile sodium chloride concentration in the steam would be proportional to the square of the ionized salt concentration in the boiler water. Regardless of the analytical technique employed, the measured distribution ratio would be a function of the boiler water concentration and not a constant.

The authors do not wish to imply that Eq 4 is the mechanism by which sodium chloride is volatilized. The data collected thus far are not sufficient to justify such a conclusion. However, it is possible that an intermediate product is involved in the vaporization process.

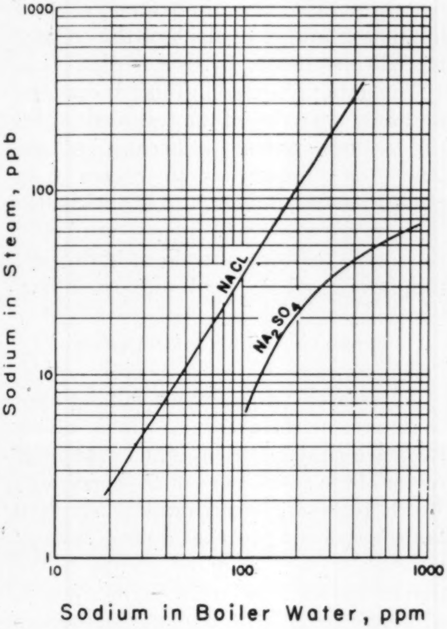

FIG. 6.—Vaporous Carry-Over of Sodium Salts at 2550-psig Salt Injection Tests.

Volatilization of silica from alkaline boiler water is generally attributed to the formation of silicic acid by hydrolysis. The dependency of vaporization upon this intermediate product causes the carry-over of silica to be a function of the pH of the boiler water.

The distribution coefficients obtained for sodium sulfate (Table VI) in the salt injection tests varied somewhat with concentration; however, they do not show the same pattern as do the results for sodium chloride. For comparison purposes, data from both tests are plotted together in Fig. 6. The vaporous carry-over of sodium sulfate was always much lower than that for sodium chloride. This, of course, was expected.

The lack of agreement between our field data and the results obtained by others in the laboratory was of concern to the authors throughout the test program. Several possible explanations were considered. The possible influence of concentration on distribution ratios is significant since there is a question as to whether adequate laboratory tests have been performed at the low concentrations that are typical in high-pressure boilers. Although both Styrikovich (1) and Sastry (3) indicate that their tests were performed over a range of concentrations, no actual data were presented in the translations which were available. Neither Straub (4) nor Morey (5) performed volatility tests in the low-concentration range.

The steam-water dynamics in a boiler were compared with those in a simulated test vessel to evaluate any fundamental differences. Introduction of feedwater into a boiler results in subcooling in the drum. The degree of subcooling depends upon the temperature of the feedwater, the circulation ratio, and the extent of mixing between the feedwater and the boiler water. In a large high-pressure unit, the mean temperature of the water in the drum might be as much as 25 F below saturation. The temperature of the water in contact with the steam phase would, of course, be somewhat higher than the average. It is difficult to predict what influence this factor might have on vaporous carry-over. The water in contact with the steam during generation and separation is obviously at saturation temperature, and the degree of contact between the steam and the water in the drum is questionable. However, a few degrees of temperature could have a significant effect on salt volatility, and the vaporous carry-over might be less in an operating boiler than in a model steam generator where the boiler water is maintained at saturation temperature.

Strictly on the basis of the data obtained in the field, vaporous carry-over does not appear to present a problem in the 2500 to 2600-psi pressure range provided reasonable limits are placed on boiler water concentrations. In the boilers tested, the total carry-over of sodium was relatively low even with higher-than-normal concentrations in the boiler water. Data collected under these conditions indicated a distribution ratio for sodium chloride which was definitely less than 5×10^{-4} and probably less than 2×10^{-4}. Converted to a more familiar form, these figures represent 0.05 per cent and 0.02 per cent carry-over, respectively. Although primary emphasis was placed in the study of sodium chloride characteristics, sodium phosphate and sodium hydroxide were present in many of the boiler water samples tested. Variations in these constituents did not appear to affect the over-all pattern of low vaporous carry-over. It is difficult to extrapolate the data obtained to predict conditions in boilers operating at higher pressures. If distribution ratios are exponential functions as many investigators believe, the magnitude of vaporous carry-over could increase rapidly with only a moderate increase in pressure.

Vaporous carry-over can be expected to complicate the determination of steam purity in high-pressure boilers. If volatile carry-over is significant, it will be virtually impossible to measure accurately the moisture content of the steam by chemical means. Where sodium analysis is employed to determine solids content, considerable question may exist as to what anions, if any, are also present in the steam. Vaporous carry-over may also present a problem in steam sampling. A small amount of condensation may absorb a large percentage of the vaporized solids in a steam sample. The sample line should be designed to ensure that any condensate formed cannot drain back into the drum. The total sample leaving the drum should be collected for analysis. No portion should be bypassed; otherwise, the sample collected might contain all or none of the concentrated condensate.

The data presented in this paper represent only a portion of the test work which the authors intend to perform on the study of "Impurities in Steam from High-Pressure Boilers." It is anticipated that future work will include studies of the characteristics of sodium hydroxide, sodium phosphate, and sodium sulfate.

SUMMARY AND CONCLUSIONS

A research program was undertaken to determine the extent to which sodium salts are subject to vaporous carry-over under high-pressure boiler conditions. Field tests were performed on three boilers operating in the 2500 to 2600-psi pressure range. Primary emphasis was placed in the study of sodium chloride, although other salts were evaluated to a lesser degree.

The results obtained indicate that the volatility of sodium chloride is relatively low at these pressures as long as the salt concentration in the boiler water is maintained within reasonable limits. With 10 to 25 ppm of sodium chloride in the boiler water, calculated distribution ratios averaged less than 2×10^{-4}, or 0.02 per cent carry-over. Under these conditions, vaporous carry-over would result in the presence of approximately 1 ppb sodium in the steam. On this basis, vaporous carry-over of sodium salts does not appear to present a serious problem with regard to turbine deposition in the 2500 to 2600-psi pressure range.

The magnitude of the vaporous carry-over observed in the field tests was considerably less than that obtained by other investigators in the laboratory. There are several possible explanations for this lack of agreement; however, present information on the mechanics of vaporous carry-over is inadequate to permit evaluation of all potential factors.

The authors plan further research work with regard to carry-over in high-pressure boilers. Future tests will include studies of the characteristics of sodium hydroxide, sodium sulfate, and sodium phosphate.

Acknowledgment:

The authors wish to acknowledge the excellent cooperation extended to them by the personnel of the Cincinnati Gas and Electric Co., the Virginia Electric and Power Co., and the Cleveland Electric Illuminating Co. These utilities not only donated the necessary test sites, but provided help and encouragement without which the test program could not have been completed.

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(3) V. Sastry, "The Solubility of Salts in High Pressure Steam with Regard to Salt Deposition in Turbines," *Chemiker-Zeitung*, Vol. 82, No. 13, July 10, 1958, pp. 456–457.

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DISCUSSION

Mr. L. E. Johnson[1] (*in written form*).—The paper by Messrs. Ulmer and Klein is very timely and appropriate. Because of the trend toward higher pressures in boilers, vaporous carry-over has assumed a dominant role in steam purification considerations.

At our Research Center we have also been measuring vaporous carry-over, and we have noted a sensitivity to pressure considerably greater than that observed by Messrs. Ulmer and Klein. Although we are in agreement with their values in the 2500 to 2600-psi pressure range, the values given in the paper for 1500 psi are 30 to 100 times greater than our observed values. At 2900 psi our measurements are appreciably higher than would be indicated if one were inclined to extrapolate the authors' data which were limited to 2600 psi.

In view of these differences, a description of our apparatus may be in order. Figure 7 shows a diagram of our test facility. It consists essentially of a 60-in. inside diameter spherical drum with a downcomer and a centrifugal pump to circulate water; a steam discharge line and a centrifugal pump to circulate steam; a mixer section to mix the water and steam; and a riser section to bring the steam-water mixture back to the drum. The full-size separating equipment to be tested is installed inside the drum. Both pumps have variable speed drives to control the quantity and quality of the mixture through the separating equipment. An automatically controlled natural-gas furnace is used for bringing the system up to pressure and to make up heat loss from the system. A considerable amount of auxiliary equipment is not shown. It includes a boiler water storage tank in conjunction with a system for controlling drum water level; an automatic condensate system for providing seal water to the pump bearings; a demineralizer; and complete instrumentation for measuring pressure, steam flow, water flow, true water level, boiler-water chemistry, carry-over, carry-under, pressure drop, and other variables essential to the control of this system. The instrumentation includes a flame spectrophotometer set up for continuous analysis and recording of the sodium content of steam samples.

Thus, we have a facility which has all of the advantages of a laboratory unit and most of the advantages of a field test unit in that full-size separating components can be tested under actual operating conditions of pressure, temperature, steam flow, and water circulation rate.

The accompanying Table VII shows the chemical analysis of the normal boiler water used during the majority of these tests. These are nominal values, only, and the actual solids for each particular test may have varied considerably from this average. However, during each test, boiler-water pH and conductivity were continuously monitored and boiler-water samples were taken periodically for chemical analysis.

Figure 8 shows typical results obtained from tests of separating equipment in the apparatus just described. The results are plotted as per cent carry-over versus steam flow for each of four different pressures. The values on the ordinates are actually the ratios of sodium in the steam to sodium in the boiler water and therefore include both mechanical carry-over and vaporous carry-over of sodium. Note that at each pressure there is a minimum value of carry-over. At the lower pressures (1500 and 2000 psi) there is a distinct flat portion of the curve at low steam flows and a definite break in the curve as the steam flow increases. The lowest values on the curve are below 0.00015 per cent, which represents a sodium concentration in the steam sample of about 0.1 ppb. At the higher pressures there is a more gradual increase in carry-over with increasing steam flow, but, if the curves are extrapolated to zero steam flow, there is still a significant amount of carry-over. These minimum values at each pressure are assumed to be vaporous carry-over.

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[1] Research Engineer, The Babcock & Wilcox Co., Alliance, Ohio.

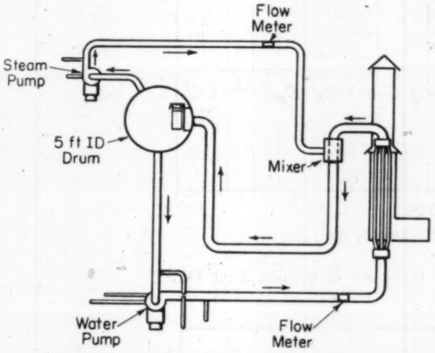

FIG. 7.—Steam Separator Test Apparatus Design Pressure 3000 psia at 695 F.

TABLE VII.—NORMAL BOILER WATER CHEMISTRY.

NaOH	30 ppm
NaCl	50 ppm
Na_3PO_4	5 ppm
Na_2SO_4	415 ppm
Total dissolved solids	500 ppm
pH	10.8

Plotted on Fig. 9 are the results of several tests of this same type. We have plotted the minimum values of carry-over (vaporous carry-over) against pressure. The heavy vertical lines represent the range of minimum values observed, and the circles represent the average of the number of points listed at each pressure. Note that our values of vaporous carry-over at 2500 psi check very closely with those observed by Ulmer and Klein. They report, in Tables I and II, average values of 0.027 per cent and 0.028 per cent at 2500 and 2600 psi, respectively, compared to our average value of 0.025 per cent at 2500 psi.

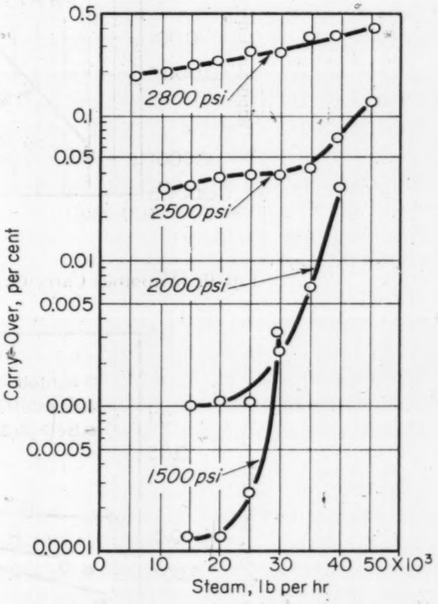

FIG. 8.—Carry-Over Versus Steam Flow at Various Pressures.

The curved line is a calculated line of vaporous carry-over of the form suggested by Styrikovich, $K = (D_s/D_w)^N$, with the exponent chosen to fit our data.

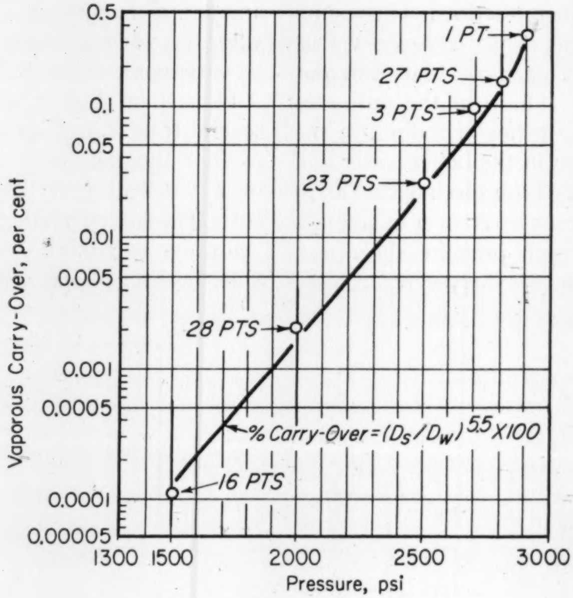

FIG. 9.—Vaporous Carry-Over—Babcock & Wilcox Test Data.

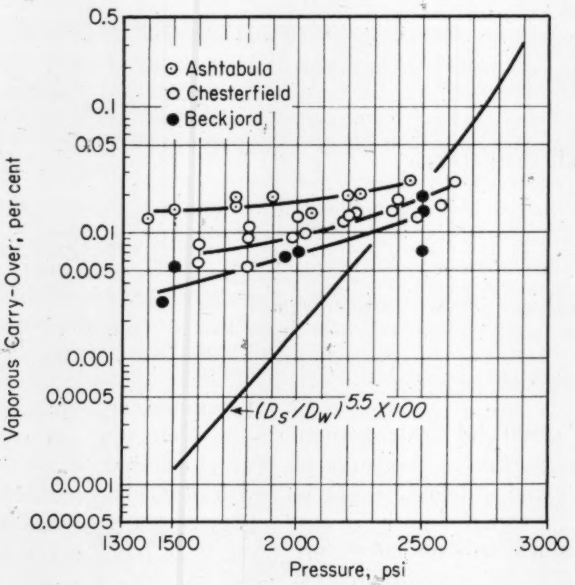

FIG. 10.—Vaporous Carry-Over—Table IV, Ulmer and Klein.

Note that a power of 5.5 fits very closely our average values. From this we have come to two tentative conclusions which must be proven by more data. First, the general form of the equation proposed by Styrikovich is correct. Second, this mixture of compounds which we had in our boiler water does not act as a mixture of several individual compounds but rather as a single complex compound. This is logical because, although this is a relatively high-solids water by high-pressure boiler standards, it is an extremely dilute solution by ordinary chemical standards, and therefore, the chemicals may be assumed to be almost, but not quite, completely ionized. Therefore, each cation present is available to combine with any anion and, consequently, the boiler water acts as a complex compound with a distribution ratio peculiar to that particular combination of compounds as we observe on Fig. 9.

Plotted on Fig. 10 are the results of the variable pressure data from Table IV of the Ulmer and Klein paper. Also shown is the curve, $K = (D_s/D_w)^{5.5}$, representing our data. Whereas all of our data, within experimental error, follow the relationship between vaporous carry-over and pressure proposed by Styrikovich, it is apparent that the authors' data do not. The values observed at 2500 psi and 2600 psi are close to our observed values. However, the values observed in the pressure range of 1400 to 2000 psi are considerably higher than we would expect.

We might offer a speculation on the reason for the difference observed. Apparently all of the data reported on Table IV was obtained in small pressure chambers connected by tubing to steam drums which were operating in the pressure range of 2500 to 2600 psi. The pressures below 2500 psi were obtained by throttling the steam and water to the desired pressure. It is then possible that the steam and water were not in intimate turbulent contact for a sufficient length of time for the salts dissolved in the steam to come to equilibrium with the salts dissolved in the boiler water. If this explanation is correct and the authors' data are in error, an extrapolation of their curves to higher pressures, say 2800 or 2900 psi, would introduce significant errors since their values of carry-over are increasing at a much lower rate than the values for our data.

We are continuing our studies of vaporous carry-over, and we are planning a more complete report in the future.

MESSRS. R. C. ULMER AND H. A. KLEIN (*authors' closure*).—The authors wish to thank Mr. Johnson for his informative discussion and for the contribution of additional data on vaporous carry-over. We are pleased to have a confirmation of our data in the 2500 to 2600-psi pressure range. The majority of our data was collected at full drum pressure for the respective boilers, and this is the area of primary interest.

We are neither greatly surprised nor concerned over discrepancies at the lower pressures because it was realized that these values might be in error. The sodium concentrations in the steam at the lower pressures were often below the detectable limit for the analytical instrument used. It was for this reason that we did not plot the data and specifically discouraged any attempt to extrapolate to higher pressures.

From the standpoint of the operator, the discrepancies described are of no practical importance. It is true that a distribution ratio of 10^{-5} is 100 times as great as 10^{-7}. However, for all practical purposes, both values may be considered zero. With 25 ppm of dissolved salts in the boiler water, a distribution ratio of 10^{-5} would produce a sodium concentration in the steam of about 0.1 ppb. Our investigation was intended to be a power plant study under relatively normal boiler conditions. We could not, therefore, have possibly reproduced the low-pressure data which Mr. Johnson presents. This would have required analysis of sodium to the nearest 0.01 ppb.

Mr. Johnson suggests that failure to reach equilibrium might have been responsible for our inability to obtain the expected zero sodium values in the reduced pressure tests. This is a logical conclusion in view of the fact that the contact time between throttling and separation was quite short. Lack of knowledge on rate of mass transfer under these conditions makes it impossible to confirm or disprove this assumption.

The fact that Mr. Johnson's data agreed with the Russian equation is noteworthy since this equation seems sound on the basis of silica volatilization data. The choice of a 5.5 exponent agrees well with our most reliable data. Particular care must be taken, however, in any theoretical analysis of vaporous carry-over. The volatile material may not be the ionized salt as we know it but rather some intermediate product activated under boiler conditions. Interaction between the ions of a solution may affect the mutual volatility of different salts. From this standpoint, distribution ratios may be constant only for a given specified solution.

The test facility described by Mr. Johnson does indeed have excellent possibilities for the study of vaporous carry-over. We look forward to the publication of his complete report. The difficulties inherent in testing with small model boilers cannot be overemphasized. Very low steaming and sampling rates make it difficult if not impossible to rinse sample bottles and glassware in an acceptable manner to perform the sensitive chemical tests required. A new fullsize test boiler designed for operation up to 3200 psig is presently being built in our research and development laboratories. We expect to perform complete vaporous carry-over studies on this unit as soon as it is available

GENERAL DISCUSSION

MR. R. W. LANE[1] (*presented in written form*).—Since the vaporous carry-over of boiler water salts from boiler water has been shown to be stoichiometric with respect to the cation and anion involved, the application of the hydrogen exchange technique should prove satisfactory for the measurement of vaporous carry-over as well as mechanical carry-over. Since the conductivity values of boiler water salts in acid form at atmospheric boiling water temperature have been determined[2] and since measuring and recording at boiling water temperature provides increased accuracy and sensitivity, the conductivity method may now be used to measure and determine actual boiler water carry-over as low as 10 to 20 ppb.

The reported technique for sampling superheated steam is a worthwhile contribution. This technique makes possible the accurate determination of steam purity of superheated steam, which previously could not be obtained.

We would like to comment on the desirability of using a pump to meter the injected water since the valve adjustment to provide natural circulation might be expected to provide inexact dilution.

The accompanying Fig. 1 shows the conductivity-pH relationship between morpholine[3] and carbon dioxide at 25 C which we feel will be of interest to the group. As equilibrium constants and equivalent conductances become available for other amines and at the higher temperatures, further graphs will be prepared.

MR. E. E. COULTER (*author*).—Mr. Lane mentioned that he would prefer to use a metering pump rather than the natural circulation system on the recirculation nozzle.

I think the choice would depend on the type of test for which the nozzle is to be used. If the test is expected to be rather short, it is much more convenient to use the pump. Certainly it can be controlled more accurately, it is easier to hook up, and the rate of recirculation can be determined more easily. In all of our tests, we have used a pump. However, if one were going to use such a sampling system for monitoring steam purity over a long period of time, the pump would be quite troublesome, and I think then it might be worthwhile setting up the natural circulation system.

MR. H. A. KLEIN (*author*).—In view of the effect of ammonia on chloride volatility the question has been raised as to possible interference of ammonia in our vaporous carry-over tests. Since our tests were run in the field, we had to contend with the ammonia normally in the steam in each particular plant. Ammonia concentrations on the average were about 0.3 ppm which, on the basis of stoichiometry, would be capable of tying up about 600 ppb of chloride. Our carry-over measurements were based solely on sodium analysis so we could not detect chloride carry-over in the form of ammonium chloride. We had to assume that the presence of ammonia did not influence the vaporization of sodium salts as such.

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[1] Chemist, State Water Survey Div., Urbana, Ill.

[2] R. W. Lane, C. H. Neff, and T. E. Larsen, *American Power Conference*, Vol. XXIII, pp. 550–558 (1961).

[3] A. R. Ingram and W. F. Luder, "The Ionization Constant of Morpholine in Water," *Journal*, Am. Chemical Soc., Vol. 64, pp. 3043–3044 (1942).

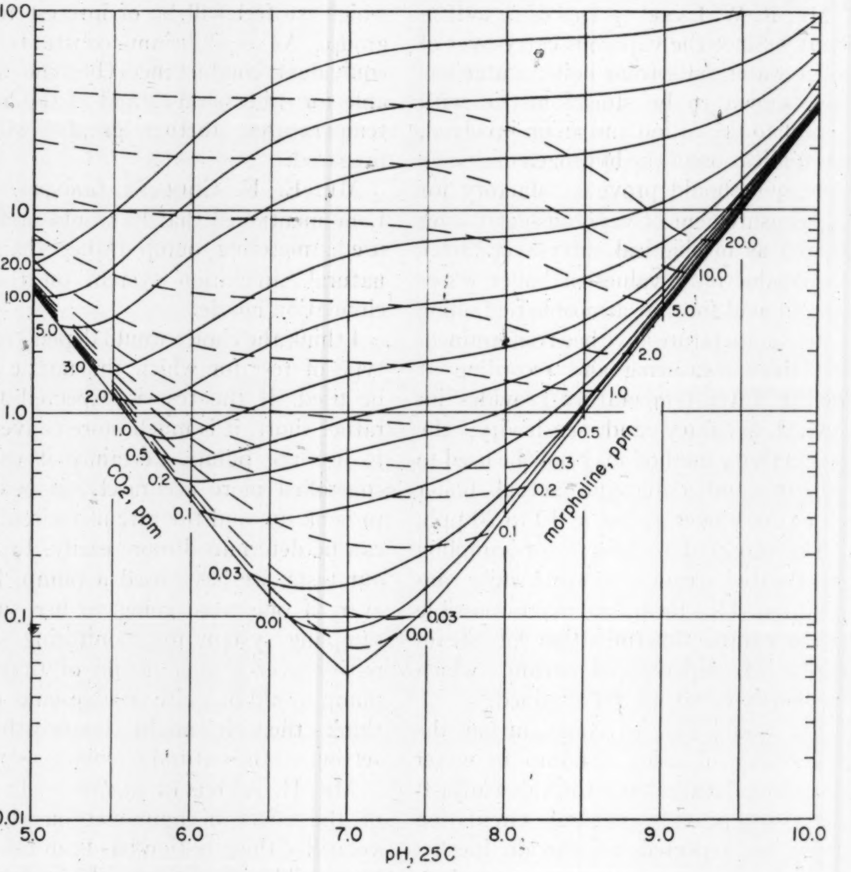

FIG. 1.—Conductivity-pH Relationship Between Morpholine and Carbon Dioxide at 25 C.

MR. R. C. ULMER (*author*).—Ammonia is another variable that is very difficult to control. We gave some thought to the possibility that ammonia might have had something to do with the results, but sufficient data were not available to draw conclusions.

I would like to be sure that everyone realizes that there was considerable difference in concentration between our tests, most of which were around 25 ppm total solids, and Styrikovich's work which was around 500 ppm. Most of the work that was done in the ASTM project was at an even higher range.

The intriguing thing is that on varying the concentration, around 500 ppm, we check the distribution ratio very closely. If we go much below that, we do not check.

I am of the opinion that perhaps we have stumbled onto something that some of the other people have missed in that they didn't vary concentration. Time will tell.

MR. M. D. BAKER.[4]—The papers as they were presented are quite interesting and worthwhile. They remind me of some things that happened in the period from 1927 to 1930. This was when steam qualities were determined with a throttling calorimeter. The main reason for mentioning it is that following a series of steam quality tests an inspection was made of a superheater inlet header. The inspection showed an appreciable amount of sodium hydroxide in the header. The boiler water constituents had a ratio of 15 parts phosphate, 5 to 10 parts sulfate, and 1 part hydroxide. Yet, the deposit found in the header that contained a large number of ferrules inserted to obtain better steam distribution was more than 90 per cent sodium hydroxide.

The fact is that the deposits found in the superheater header were placed there with reasonably high velocities of the steam. Experiments reported in the papers gave results of vaporous carry-over with practically zero velocity of the steam. When the amount of steam produced by the boiler was reduced, the velocity of the steam through the purifying equipment was reduced. As a result, the deposition of sodium hydroxide disappeared. The thought is that possibly the amounts of volatilization products produced would be greater if it were possible to increase the flow of steam from the surface of the test solution. A more rapid removal of the vapor without producing physical carry-over should be used before the reported figures and theories are accepted as final.

MR. E. B. MORRIS.[5]—With respect to Mr. Rubright's paper, he makes two statements. One is an assumption that since this particular test boiler was used for the silica carry-over studies it had been proven that it was not subject to mechanical carry-over.

The second statement was that since they used a range of 2 to 5-lb per hr flow in these tests, mechanical carry-over did not exist.

I am wondering, because I do not recall how it was first determined, that this test facility was not subject to mechanical carry-over; when you put 5000 ppm sodium chloride in such a device you change the entire boiling characteristics of the water.

In Ulmer and Klein's paper, Fig. 6 shows the parameters, or the relationship, between sodium chloride in the boiler water and sodium chloride in the steam. I note that for a ten-fold concentration in the boiler water there is a 50-fold concentration in the steam. This is attributed to the change in distribution ratio.

On a practical basis, we started up a 2000-lb pressure boiler about 3 yr ago on conventional sodium treatment. The total solids probably never exceeded 100 ppm, and the chloride content never exceeded 1 ppm. But in short order, the boiler feed pump turbine which was supplied with steam from the exhaust of the high-pressure turbine fouled up. It was opened and deposits were found consisting of boiler water salts including sodium chloride. This fouling occurred repeatedly as demonstrated by the opening of the high-pressure valve, or the by-pass valve.

Then the boiler water conditioning was changed to a modified low solids treatment, the solids being limited to 25 ppm, although they probably never approached that. The volatile treatment was ammonia with about 5 ppm phosphate maintained. After almost a year's operation the feed pump turbine was again opened and found completely clean. Now all this leads to the question:

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[4] Chief Chemist, West Penn Power Co., Springdale, Pa.

[5] Head, Chemical Section, American Electric Power Service Co., New York, N. Y.

Is there perhaps a limiting concentration of impurities in the steam which will cause deposits in turbines rather than a linear function? If a linear function exists, 10 ppm would build up deposits at a certain rate. If you have a hundred ppm, it would build up ten times as fast. That is my specific question.

In connection with the Cobb and Coulter paper, we observed in 1938 a 1300-psi boiler showing a consistent difference in conductivity of saturated and superheated steam. The saturated steam showed twice the conductivity as the superheated, even though the saturated steam was only a half a micromho. In those days, this was attributed to lack of isokinetic sampling. However, I think we perhaps now have the true cause.

I think if we let our imagination soar a little, perhaps this mechanism of deposition in superheated steam sampling lines from the vapor phase could be a possible avenue of approach, or a technique, to study further the mechanism and the magnitude of vaporous carry-over.

MR. KLEIN.—Mr. Morris has referred to the salt injection tests which we performed at Ashtabula Station to study the influence of solute concentration on the volatility of salts at high pressure. Mr. Morris is apparently concerned that the increased carry-over of sodium chloride that we observed with high concentrations might have been due to increased mechanical carry-over.

High salt concentrations in boiler water can affect the surface tension of the water and under severe conditions will promote foaming. Such phenomena are, however, generally associated with high-load operation where agitation is great. The steam rise velocities through our test chamber were on the order of 0.2 in. per sec, which would seem to be sufficiently low to permit complete separation. Furthermore, if mechanical carry-over were responsible for the salt in the steam, we would have expected to observe increased carry-over with increased steam velocity.

In the paper we discussed a theory which might possibly explain the apparent variation of the sodium chloride distribution ratio with different salt concentrations. This is only a theory, however, and considerably more experimentation will have to be performed before any sound evaluation of the data can be made.

MR. M. M. RUBRIGHT (*author*).—The mechanical carry-over characteristics of this test boiler were determined during Coulter's work on silica carry-over. The fact that there was a very small amount of mechanical carry-over at the flow rates mentioned—5 lb per hr, or less—was determined using phosphorus-32 as a tracer. The isotope was put in the boiler during the silica test. Assuming that phosphorus has practically no vaporous carry-over relative to the silica, if there were mechanical carry-over there would be phosphorus-32 in the condensate. The phosphorus-32 content in the condensate was very, very small.

In these tests, we were a little worried about this ourselves. It was possible that minor test changes could have produced changes in boiler characteristics or that we may have changed its performance characteristics by the relatively high salt concentration used. We found no apparent difference in the magnitude of the carry-over obtained at 2 lb per hr and at 5 lb per hr. This should have increased somewhat at the 5-lb rate if there were mechanical carry-over.

It was also planned that our system should be, insofar as possible, a stagnant system as far as mechanical upheaval of the boiler water surface itself was concerned.

Our retention time was quite long in this boiler. We again deliberately chose this condition, but it gave us a few difficulties later on. Our retention time was in the order of about 1 hr for a 2-lb per hr steam flow rate. It was reduced by a factor of $2\frac{1}{2}$ in the 5-lb per hr test. We assumed that this long retention time would tend to produce conditions closer to equilibrium.

We have used these facts as a basis for the belief that we were not measuring mechanical carry-over—the fact that we had no drastic increase in our carry-over when the steam production rate was increased from 2 to 5 lb per hr, the stagnant conditions in the boiler, and the long retention time.

We did not originally plan on using a concentration as high as the 5000 ppm we finally used in this test. We originally planned to use 250 ppm, because we were relying at that time on the magnitude of carry-over as published by the Russians. We used 250 ppm in the first few tests we ran and found that we could not analyze the condensate at all for sodium or chloride content, so we took the drastic step of increasing our concentration by a large factor.

We are fairly sure the carry-over we measured was vaporous rather than mechanical.

CHAIRMAN J. K. RICE.[6]—Are there any other comments covering some of the things Mr. Morris mentioned. There were two matters that he brought up:

1. The possibility of conducting further investigations on deposition of salts from steam, taking advantage of the deposition noted in the superheated steam sampling line as a tool for study, and

2. At what concentration will solids come out of steam; if one concentration causes a certain amount of deposition will ten times as much cause ten times as much deposition?

I think part of what Mr. Morris is driving at can be answered if you consider that the substances are generally in true solution in steam; that it will probably be seldom in modern high-pressure temperature operation that the substances are present as dust. Styrickovich, some time ago, gave rather complete data on sodium sulfate solubility in superheated steams at various pressures and temperatures. This data showed that sodium sulfate reaches minimum solubility in steam at a point 50 to 70 C above saturation. This 50 to 70 C point of minimum solubility for sodium sulfate has, in my own experience, agreed very well with the observed sodium sulfate deposition in superheaters. Such deposition occurs in the first passes of superheaters and over a limited length of the superheater; it usually disappears altogether by the outlet end. The point of maximum deposition corresponds reasonably well with the point of minimum sodium sulfate solubility. Deposition in a superheater is apparently quite selective.

The common boiler water salts have individual solubilities in steam; some of them may have retrograde solubility, at least near saturation. In the turbine itself, if you will think of deposition as a dynamic crystal growth process as well as a solubility-saturation problem, it seems a certain amount of supersaturation could occur before deposition began. The actual point of saturation in the steam may be well up near the turbine inlet. It may take a relatively long time, measured in milliseconds, to actually begin to nucleate. All these things must be considered. When I introduced this symposium, I said that I think we know absolutely nothing of the dynamics of deposition in steam turbines. I think this is one of the problems that would be encountered in trying to study deposition by means of a capillary sampling tube.

MR. H. M. RIVERS.[7]—First, with

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[6] Consulting Engineer and President, Cyrus Wm. Rice & Co., Pittsburgh, Pa.

[7] Assistant Director, Hall Laboratories, Div. of Hagan Chemicals & Controls, Inc., Pittsburgh, Pa.

reference to the Cobb and Coulter paper, it is apparent that the authors have demonstrated, explained, and, to a realistically practical extent, solved the problem of solids deposition in superheated steam sampling lines. Those of us who must on occasion determine the purity of high-pressure, high-temperature steam will be quick to recognize the value of this new sampling technique—and take advantage of it.

When we are dealing with volatilized contaminants in steam, any one of several types of sampling nozzles will probably give reliable samples. But some knotty questions remain to be answered regarding the validity of steam samples when solids may be present in something other than vaporous form. For example, if superheated steam is contaminated with solids dissolved or suspended in droplets of boiler water, do these solids completely volatilize in the superheater? If not, are the solids converted to extremely fine particles of dust, and are these particles distributed homogeneously as is necessary for accurate sampling of the steam? And how can we always be sure of getting a representative sample of steam in the face of strong evidence that solids may sometimes be carried in highly concentrated liquid flowing along the surface of a superheater element or superheated steam line?

These questions are pertinent to all three of the papers under discussion. Some day we shall know what kinds and amounts of volatilized solids contaminants we should expect in steam produced from boiler water of known composition at a given temperature. Then if the steam is found to be contaminated in any way other than what we should expect, we shall have a realistic basis for appraising boiler performance. But steam purity tests of this nature will be meaningless if the condensed steam subjected to analysis is not of the same composition as the original steam at the point of sampling. In this connection, one could hardly overemphasize the importance of preserving the integrity of steam samples while on their way to the analytical apparatus.

Twenty years ago, a German investigator, F. Spillner, studied the volatility of sodium chloride in high-pressure superheated steam and reported as follows: "Before everything else, the fact that the concentration of the salt in the steam is independent of the salt concentration of the solution over wide limits, must be considered to be remarkable." Remarkable indeed! Actually, we know now that some of the mineral components in high-pressure boiler water volatilize in a way that is very definitely related to the composition of the boiler water.

Obviously, of course, there would be little point to investigations like those under discussion if it were possible to maintain boiler water absolutely devoid of solid contaminants. Industrial plants and central stations being what they are, however, it will probably be a long while before we lose serious interest in the volatility of boiler water solids introduced as trace contaminants in the boiler feedwater.

As long as we have water in steel boilers, we are going to have iron oxide in the boiler water. Can any of this iron oxide leave the boiler as a volatile component in the steam? Many of you, surely, have noticed how rapidly iron oxide may accumulate on the glass parts of cells used in measuring the conductivity of condensed high-pressure steam. This has been observed even where sample lines are made of stainless steel. We have found significant amounts of iron oxide and—believe it or not—copper in high-pressure turbines. Many of us are curious about these indications that iron oxide and copper are volatile, at least to some slight extent, in high-pressure steam. Should anybody worry about this? Is it something that warrants serious investigation?

MR. R. C. ULMER (*author*).—I certainly agree with Mr. Rivers that we are going to have to give a lot of consideration to the possibility that such things as copper and iron volatilized in steam. In fact, with our little supercritical pressure boiler in Chattanooga we are right now setting up some contaminator tests, as we call it, where we take a pot of clay type vessel and have pure copper in one of them, and we have copper oxide of two different types in two of the others. We are going to put steam through these at a very low flow and determine the amount—we shall call vaporized copper and vaporized iron—and then determine the amount of these constituents in the steam. I am sure everybody knows there have been three or four turbines that have been pretty badly contaminated with copper. So it appears that copper is volatilized, and in answer to Mr. Rivers' suggestion we are going to try to find out how much actually does vaporize.

MR. F. E. CLARKE.[8]—It was considerate of the Chairman to place the Cobb and Coulter paper last in the program so that sampling errors discussed therein would not embarrass the other speakers. It is disturbing to think of the useless man-hours chemists have spent analyzing samples and the countless arguments which such data must have generated among plant personnel and statisticians. It is a sad fact that the importance of sampling has been minimized too frequently. All of us could profit by a little more scientific thinking where sampling is concerned.

Mr. Rubright's discussion of stoichiometry in steam contaminants was very interesting. The concept of preferential carry-over of ions is a bit difficult to grasp. The molecular carry-over pointed up by this study is more easily understood and more generally acceptable. I was a bit puzzled that Mr. Rubright deleted questionable data from his first series, while including obviously abnormal values in the second series. Perhaps he was influenced by a statistician.

The paper by Ulmer and Klein inadvertently explains apparent inconsistencies in steam quality data. Dr. Robert Rosenthal recently remarked that looking for the concentrations of solutes these authors discuss is like looking for one person in the population of the earth (0.5 ppb). The differences between the data of Johnson and those of Ulmer and Klein become rather insignificant in the light of this example, particularly if one considers the sampling errors discussed by Cobb and Coulter. When reservations are made for these factors, the effects of ammonia, etc., it is evident that all of the authors are talking about essentially the same order of steam quality.

Mr. Lane made a pertinent observation that stoichiometry in steam contaminants facilitates use of simpler methods for steam analysis. His interesting graph for CO_2 and morpholine correction will be useful to all concerned with steam quality.

There are no logical answers to the steam quality puzzle posed by Mr. Baker. Maybe his operator wasn't really putting into the boiler what he thought.

Mr. Rivers gave us something new to think about. This is good, since a research man must never run out of ideas. If copper carry-over can be confirmed, or refuted, there always will be new problems like the recently reported deposition of titanium compounds in a power station boiler. A man's reach must exceed his grasp if we are to have future symposiums.

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[8] U. S. Geological Survey, Water Resources Div., Quality of Water Bureau, Washington, D. C.

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON MAJOR EFFECTS OF MINOR CONSTITUENTS ON THE PROPERTIES OF MATERIALS

The effects of impurities in low concentrations is a subject of increasing importance in all engineering fields. This Symposium on Major Effects of Minor Constituents on the Properties of Materials was organized to treat the subject broadly, bringing together a physicist, a chemist, a ceramist, and a metallurgist, and weaving the thread of impurity effects through crystalline solids, liquids, and ionized gases. It was apparent in organizing this symposium that purity means something different in each branch of science. Bringing out this divergence of viewpoint in different materials areas was one of the accomplishments of the symposium.

This Symposium was sponsored by the ASTM Division of Materials Sciences, and the papers were presented at the third and sixth sessions of the Sixty-fourth Annual Meeting of the Society, on Monday, June 26, 1961, at Atlantic City, N. J. Co-chairmen of the Symposium Committee and presiding at the two sessions were I. V. Williams, Bell Telephone Laboratories, and John D. Sullivan, Battelle Memorial Inst.

The papers included in the symposium are:

Impurity Effects in High-Purity Metals—L. L. Wyman and G. A. Moore
The Effect of Impurities on the Properties of Ceramic Materials—Ivan B. Cutler
Major Effects from Minor Constituents—Role in Organic Structural Polymers—John F. Lontz
The Problem of Gas Purity in Plasma Physics and Technology—S. J. Buchsbaum and G. A. Sawyer
Effects of Small Amounts of Extraneous Materials on Properties of Petroleum, Petroleum Products, and Related Liquids—Harold M. Smith

These papers with discussion were issued as *ASTM Special Technical Publication No. 304*, entitled "Symposium on Major Effects of Minor Constituents on the Properties of Materials."

SUMMARY OF PROCEEDINGS OF SYMPOSIUM ON RADIATION EFFECTS IN REFRACTORY FUEL COMPOUNDS

This Symposium on Radiation Effects in Refractory Fuel Compounds presents the latest data available on the effects of radiation on the properties of refractory fuel compounds as well as discussion of some of the newer fuel compounds.

The Symposium, sponsored by ASTM Committee E-10 on Radioisotopes and Radiation Effects, included three sessions of papers and a panel discussion. The Symposium was held during the eleventh, fourteenth, nineteenth, and twenty-first sessions of the Sixty-fourth Annual Meeting of the Society, June 27–28, 1961.

Mr. John E. Gates, Battelle Memorial Inst., served as Symposium Chairman and presided over the twenty-first session. Messrs. Ersel A. Evans, Hanford Atomic Products Operation, General Electric Co., and J. B. Trice, Aircraft Nuclear Propulsion Division, General Electric Co., presided over the eleventh and fourteenth sessions, respectively, while Mr. M. J. Whitman, U. S. Atomic Energy Commission, was moderator of the Panel Discussion held during the nineteenth session.

The papers included in the Symposium are:

Some Consequences of Excess Oxygen in UO_2—J. A. L. Robertson
Voids in Irradiated UO_2—J. Lambert Bates
The Heat Rating Required to Produce Central Melting in Various UO_2 Fuels—A. S. Bain
Fission Fragment Tracks in Thin Films of UO_2—T. S. Noggle and J. O. Stiegler
Effects of Burnup on Certain Ceramic Fuel Materials—M. L. Bleiberg, W. Yeniscavich, and R. G. Gray
Study of Factors Controlling the Release of Xenon-133 from Bulk UO_2—D. F. Toner and J. L. Scott
In-Pile Fission-Gas Release from UO_2—J. B. Melehan and F. A. Rough
Continuous Release of Fission Gas from UO_2 During Irradiation—R. M. Carroll
Some Comments Regarding Sintering Characteristics in a Radiation Environment—E. A. Aitken
Irradiation of BeO-UO_2 Fuel Pellets—D. E. Johnson, J. Koretsky, and A. K. Smalley
Mechanism of Irradiation Damage in Uranium Monocarbide—D. G. Freas, A. E. Austin, and F. A. Rough
Water-Vapor Corrosion of Beryllia—C. C. Browne
The Irradiation and Examination of a Plutonium-Uranium Oxide Fast Reactor Fuel—J. M. Gerhart, J. N. Siltanen, and J. S. Cochran
Panel Discussion

These papers with discussion, with the exception of the paper by D. E. Johnson, J. Koretsky, and A. K. Smalley, were issued as *ASTM Special Technical Publication No. 306*, entitled "Symposium on Radiation Effects in Refractory Fuel Compounds."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON EROSION AND CAVITATION

The subject of erosion and cavitation is very important in hydraulic turbines, diesel engines, and many other applications. Recognizing the need for further study of the phenomena in these fields, the Administrative Committee on Simulated Service Testing decided to organize a symposium to determine the present knowledge of these phenomena.

The Symposium on Erosion and Cavitation, sponsored by the Administrative Committee on Simulated Service Testing, was held on Thursday, June 29, 1961, during the thirty-third session of the Sixty-fourth Annual Meeting of the Society at Atlantic City, N. J.

Mr. R. E. Peterson, of Westinghouse Research Laboratories, Pittsburgh, Pa., acted as Symposium Chairman and presided over the session.

The papers included in the Symposium are:

Introduction—R. E. Peterson
Impact of Liquid Drops—Olive G. Engel
Cavitation in Hydraulic Turbines—William J. Rheingans
Erosion by Liquid Impact—S. M. DeCorso and R. E. Kothmann
Accelerated Cavitation Erosion and Sand Erosion—W. C. Leith and W. S. McIlquham
Erosion by Solid Particles in a Fluid Stream—Iain Finnie
Deformation of Solids by Impact of Liquids at High Speeds—J. H. Brunton

These papers with discussion were issued as *ASTM Special Technical Publication No. 307*, entitled "Symposium on Erosion and Cavitation."

SUMMARY OF PROCEEDINGS OF SYMPOSIUM ON CURRENT RESEARCH ON MOTOR GASOLINE WHICH MAY AFFECT FUTURE SPECIFICATIONS

The Symposium on Current Research on Motor Gasoline Which May Affect Future Specifications, sponsored by Technical Committee A on Gasoline of ASTM Committee D-2 on Petroleum Products and Lubricants, was held during three sessions on February 7, 1961, in Philadelphia, Pa., in connection with the meeting of Committee D-2.

Mr. J. B. Rather, Socony Mobil Oil Co., Inc., served as Chairman of the Symposium Committee. Mr. H. M. Smith, U. S. Bureau of Mines, Bartlesville, Okla., chairman of Committee D-2 and Technical Committee A, presided over the morning session. Mr. D. P. Barnard, formerly of Standard Oil Co. of Indiana, presided over the afternoon session, while Mr. Rather presided over the evening session.

The papers included in the Symposium are:

Introduction—Harold M. Smith
Relation Between Laboratory Knock Ratings and Customer Satisfaction on the Road—J. E. Getz and T. O. Wagner
Other Abnormal Combustion Phenomena—Are Gasoline Specifications Necessary?—D. L. Pastell and K. Hyatt
CRC (Coordinating Research Council) Work on Antiknock—H. J. Gibson and H. I. Wilson
Motor Gasoline Octane Test Method Developments—F. C. Burk
The Role of Motor Gasoline Additives—John M. Dempster
Gasoline Stability: Possible Effects of Current Research on Future Methods and Specifications—R. O. Bender
The Effect of Composition and Engine Requirements on Future Motor Gasoline Specifications—W. J. Faust
Significance in Evaluation of Motor Fuel Volatility—T. W. Legatski and O. C. Bridgeman
Present and Future Requirements of Motor Gasoline to Meet Needs for Satisfactory Engine Performance—T. H. Risk and A. E. Cleveland
Summary—Dan P. Barnard

These papers with discussion were issued as *ASTM Special Technical Publication No. 298*, entitled "Symposium on Current Research Which May Affect Future Specifications."

SUMMARY OF PROCEEDINGS OF SYMPOSIUM ON NON-NEWTONIAN VISCOMETRY

A Symposium on Non-Newtonian Viscometry, arranged by ASTM Committee D-2 on Petroleum Products and Lubricants, was held in Washington, D. C., on October 11, 1960. The papers presented were selected with the view that in collected form they might yield a fairly comprehensive view of the methods used in investigating the viscometric properties of non-Newtonian, polymer-modified liquids—a class of liquids which includes many lubricants.

Mr. Theodore W. Selby, General Motors Research Laboratories, served as Symposium Chairman and presided over the sessions. Mr. J. B. Rather, Jr., served as Secretary and Vice-Chairman.

The papers included in the symposium are:

Introduction—Theodore W. Selby
Non-Newtonian Behavior in Lubrication—W. Philippoff
Correlation Between Rheological Parameters Measured in Instruments of Different Geometries—J. G. Brodnyan, F. H. Gaskins, and W. Philippoff
Development and Application of the PRL Single-Pass Capillary Viscometer—E. Erwin Klaus, Francis M. Angeloni, and Merrell R. Fenske
Experiences with a High Shear Rate Rotational Viscometer—F. J. Bonner and J. F. Gyer
Rotating Cylinder Viscometers for Gelled Materials—R. J. Russell
Viscometry of Polymer Solutions in a Coaxial-Cylinder Viscometer Under High Rates of Shear—E. W. Merrill
Application of the Cone-Plate Viscometer to Non-Newtonian Systems—E. O. Forster and H. H. Horowitz
The Forced-Ball Viscometer and Its Application to the Rheological Characterization of Mineral Oil Systems—T. W. Selby and N. A. Hunstad
Non-Newtonian Intrinsic Viscosity of Linear Macromolecules—Anton Peterlin
An Investigation of Low-Temperature Viscosity Characteristics of Motor Oils—G. A. Kohl and R. D. Skoglund
ASTM Investigation of Low-Temperature Viscometry of Engine Oils—T. W. Selby
Appendix: Methods of Obtaining the Low-Temperature Viscosity of Engine Oils at Higher Shear Rates and Stresses

These papers with discussion were issued as *ASTM Special Technical Publication No. 299*, entitled "Symposium on Non-Newtonian Viscometry."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON MATERIALS AND ELECTRON DEVICE PROCESSING

The Symposium on Materials and Electron Device Processing, sponsored by Subcommittee X on Control of Contaminants of ASTM Committee F-1 on Materials for Electron Tubes and Semiconductor Devices, was presented on April 5, 6, and 7, 1961, during a National Technical Meeting at Philadelphia, Pa.

Members of the Symposium Committee presided at the six sessions as follows: Session 1, Air-Borne Contaminants, N. L. Hobbs, Wyeth Laboratories; Session 2, Analytical Methods and Instrumentation, F. Van Luik, General Electric Co.; Session 3, Processing Apparatus and Techniques, H. A. Stern, Radio Corp. of America; Session 4, Materials and Processing Techniques, J. Lineweaver, Corning Glass Co.; Session 5, Gaseous Contaminants, V. C. Smith, Barnstead Still and Sterilizer Co.; and Session 6, Influence of Cleaning on Device Performance, D. E. Koontz, Bell Telephone Laboratories.

Mr. Koontz served as Symposium Chairman.

The papers included in the symposium are:

The Role of Impurities in Impurity-Dominated Electronic Materials and Devices—F. J. Biondi

Air-Borne Contaminants

Particulate Concentrations in Ultraclean Rooms—F. W. Van Luik, Jr.

A Comparison of Electrostatic and High-Efficiency Impingement Air Filters for Use in Device Development Laboratories—George Helmke

Dust Monitoring by the Dry Slide Settling Technique—W. J. Whitfield, R. C. Marsh, and I. M. Kodel

An Approach to Control of Transistor Processing Environments—C. A. Smith and T. Manns

Size Distribution and Identification of Air-Borne Particulates—Fred Allison

Analytical Methods and Instrumentation

Electron Beam Microanalyzer: A New Tool for Electron Device Development—J. S. Hayes

Detection of Inorganic Contamination on Surfaces by an Emf Measurement—D. G. Schimmel

Electrode Potential: A Tool for the Control of Materials and Processes in Electron Device Fabrication. I: Emf-Time Studies of Clean and Contaminated Platinum Electrodes—D. O. Feder and E. S. Jacobs

Electrode Potential: A Tool for the Control of Materials and Processes in Electron Device Fabrication. II: Emf Studies of the Behavior of 36 Per Cent Nickel-Iron Alloy in Hydrogen Peroxide—Formic Acid Etchants—D. O. Feder, W. E. Howden, D. R. Huff, and J. W. Richards

Use of Radioisotope Tracer Techniques in Contamination Studies—T. J. Bulat

Gas-Phase Proportional Counting: A Radiotracer Technique for Examination of Electronic Components and Materials—Herman A. Stern

Processing Apparatus and Techniques

Fluorine Release from Glasses—Josef Francel

Elimination of Contaminants from Electron Tube Components: Automation of Chemical Processing—C. O. Thomas, W. H. Craft, and D. E. Koontz

Transducer Parameters Affecting Sonic Cleaning—L. J. Lyons

Removal of Water-Soluble Flux from Assembled Printed Circuit Boards—Jack E. Smallwood

Arc Sealing of Borosilicate Glasses—Morris Liebson

Recommended Practices for Safe Handling of Beryllium Oxide Ceramics—Laurence E. Ferreira

Beryllium Oxide Dielectric Heat Sinks for Electronic Devices—P. Hessinger, A. Strott, and B. Haura

Sulfamic Acid Cleaning of Electron Tube Parts—Robert W. Etter

A 24-Position Semiautomatic Vacuum Tube Exhaust Machine—L. Quaglia, J. Byrne, and G. Reyling

Purification, Distribution, and Monitoring of High-Purity Gases for Electron Device Processing—Mal D. Rogers

Gaseous Contaminants

Comparison of Standard Leak Rates Measured by Viscous and Molecular Flow Techniques—D. W. Maurer

Gas Composition During Pumping and in Sealed-Off Power Tubes—P. F. Váradi, H. A. Langer, and M. N. Saltzman

Determination of Trace Amounts of Oxygen in Protective Gaseous Ambients—D. O. Feder and W. H. Craft

Mass Spectrometric Studies of Power Klystron Exhaust Processing—Lowell Noble and Robert K. Waits

Quality Control of Getters—P. della Porta and E. Airoldi

Residual Gas Content as a Function of Tube Processing—H. B. Frost

Influence of Cleaning on Device Performance

Changes in Stability of Germanium Alloy Transistors Through Etching, Washing, and Encapsulation Processing—Arnold J. Borofsky

Effects of Various Cleaning Methods on Electronic Components in the Micromodule—J. L. Vossen

Noise-Tested Cathode Activation—F. Dlouhy and H. J. Schuetze

These papers were issued as *ASTM Special Technical Publication No. 300*, entitled "Materials and Electron Device Processing." Papers presented at the symposium but not included in the published volume were: "Use of Ultrasonics in the Cleaning of Printed Circuit Boards" by G. B. Hollerith; "Purification, Distribution, Control, and Monitoring of High-Purity Gases for Electron Device Processing" by D. T. Manus; "Outgassing Caused by Electron Bombardment of Glass" by J. Lineweaver; "Process Variables and Their Influence on the Gas Content of Anode Materials" by B. J. Bliss; and "Problems Related to Parts Cleaning and Firing" by R. P. Misra and R. R. Mix. The papers by Messrs. Allison, Rogers, and Noble and Waits listed above were not presented during the symposium but have been included in the published volume.

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON TESTING BUILDING CONSTRUCTION

Curtain wall construction has introduced new factors in the design and analysis of building construction. This has influenced the attention given to the need for proper testing of this type of construction. The Symposium includes two papers on the subject.

The papers and discussions included in this Symposium were presented at a technical session held during a two-day meeting of Committee E-6 on Methods of Testing Building Constructions, November 14–15, 1960, held in conjunction with the 1960 Fall Conference of the Building Research Inst. held at the Shoreham Hotel, Washington, D. C.

Mr. R. F. Legget, National Research Council of Canada, acted as Chairman of the symposium session.

The papers included in the symposium are:

Introduction—R. F. Legget
Performance Testing of Metal Curtain Walls—Wayne F. Koppes
Structural Testing of Clay Masonry Curtain Walls—C. B. Monk
Measurement of the Effect of Moisture on Heat Transfer Through Insulated Flat-Roof Constructions—F. J. Powell and H. E. Robinson
Loading Tests on Full-Scale House Roofs—H. J. Thorburn and W. R. Schriever

The papers were issued as *ASTM Special Technical Publication No. 312*, entitled "Symposium on Testing Building Construction."

HANDBOOK OF VAPOR DEGREASING

The Handbook for Vapor Degreasing is presented as a guide to consumers who desire comprehensive information on the vapor degreasing process. This handbook should be helpful to those contemplating the purchase of cleaning equipment to determine the applicability of the vapor degreasing process to their problem. It will also guide those currently using vapor degreasers in the use of their equipment more effectively.

Committee D-26 on Halogenated Organic Solvents, W. D. McMaster, chairman, prepared the handbook under the direct responsibility of Subcommittee II on Vapor Degreasing, V. B. Smesrude, chairman.

The Table of Contents shows the following sections:

Definition and Process Description
Applications
Selection of a Vapor Degreasing Solvent
Equipment
Operation
General Hazards of Perchlorethylene and Trichlorethylene

This publication was issued as *ASTM Special Technical Publication No. 310* entitled "Handbook of Vapor Degreasing."

TECHNICAL PAPERS PUBLISHED IN *MATERIALS RESEARCH & STANDARDS*—1961

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* Evaluation of Resistance Strain Gages at Elevated Temperatures—R. L. Bloss, p. 9; Discussion—A. M. Schwartz, Peter Stein, W. G. Bader, and G. F. Weissmann

Fracture Characteristics of Notched Tensile Specimens of Titanium and a Titanium Alloy—G. W. Geil and N. L. Carwile, p. 16

A Stainless Steel for Standard Weights—S. J. Rosenberg and T. P. Royston, p. 21

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A Simple Stress-Corrosion-Cracking Test for Copper Alloys—D. H. Thompson, p. 108

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1961 LIST OF SPECIAL TECHNICAL PUBLICATIONS

The publications listed below have been compiled during the year 1961 to be issued as separate publications not included in either *Proceedings* or *Materials Research & Standards*. For a list of earlier Special Technical Publications, see the original listings on the last page of *Proceedings*, Vol. 49 (1949) to Vol. 60 (1960). Also a complete list of Special Technical Publications covering all publications, up to and including 1950, is included in the 50-Year Index to ASTM Publications, and complete lists up to and including 1960 are published in the two succeeding 5-Year Indexes to ASTM Publications covering the years 1951–1955 and 1956–1960.

Special Technical Publication Number	Title
56-O	1959 Supplement to the Bibliography and Abstracts on Electrical Contacts (1961)
288	Bibliography and Abstracts on Thermostat Metals (1961).
296	Physical Properties of Metals and Alloys at Cryogenic and Elevated Temperatures (1961).
297	A Five Attribute System of Describing Visual Appearance—D. B. Judd (1961).
298	Symposium on Current Research on Motor Gasoline Which May Affect Future Specifications (1961).
299	Symposium on Non-Newtonian Viscometry (1961).
300	Symposium on Materials and Electron Device Processing.
301	Symposium on Fire Test Methods (1961).
302	Symposium on Evaluation of Metallic Materials in Design for Low-Temperature Service (1961).
303	Symposium on Elevated-Temperature Compression Testing of Sheet Materials (1961).
304	Symposium on Major Effects of Minor Constituents on the Properties of Materials (1961).
305	Symposium on Soil Dynamics (1961).
306	Symposium on Radiation Effects in Refractory Fuel Compounds (1961).
307	Symposium on Erosion and Cavitation (1961).
308	Symposium on Extension of Sensitivity for Determining Various Constituents in Metals (1961).
309	Papers on Road and Paving Materials and Symposium on Microviscometry (1961).
310	Handbook of Vapor Degreasing (1961).
311	Evaluation of Compression Set Testing—Standard vs. Variable Deflection Methods—W. H. King and R. E. Harding (1961).
312	Symposium on Testing Building Construction (1961).